



**Ministry of Environment,
Lands and Parks**

**BRITISH COLUMBIA MINISTRY OF THE
ENVIRONMENT, LANDS, AND PARKS
PETROLEUM HYDRDROCARBON METHODS STUDY (1998)
ROUND ROBIN AND SINGLE LABORATORY RESULTS
FINAL REPORT**

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Prepared for:

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EXECUTIVE SUMMARY

In July of 1998, private and government laboratories in British Columbia and Alberta were requested by the British Columbia Ministry of the Environment, Lands, and Parks (BCMELP) to participate in a Petroleum Hydrocarbon round robin quality assurance study. The purpose of the study was to evaluate the performance and associated variability of BC Environment's new analytical methods for Petroleum Hydrocarbons (Draft 2.0, October 1998).

Nine laboratories participated in the round robin study, which evaluated the new hydrocarbon methods using eight Reference Samples containing petroleum hydrocarbons, and seven Quality Control (QC) samples that are daily QC requirements specified by the methods. Where applicable, Polycyclic and Monocyclic Aromatic Hydrocarbons (PAH and MAH) were evaluated in addition to the aggregate hydrocarbon parameters described in the methods. Replicate analyses ($n \geq 6$) were performed by ASL Analytical Service Laboratories for the same fifteen samples, and are included in this report as Single Laboratory Data.

Overall reproducibility of results obtained for both the round robin and the single laboratory components were very good. Comparative equivalency was excellent between the means of the round robin and single laboratory data.

For the eight Reference Samples, the round robin precision for the hydrocarbon parameters was surprisingly good, returning relative standard deviations ranging from 9.1-29.5%. This degree of variability was significantly better than the round robin precision seen for the PAH and MAH target compounds (an unexpected finding). As expected, single laboratory data was considerably more precise than round robin data. For the petroleum product Reference Samples, methodological accuracy could not be directly evaluated, since the "true values" for the hydrocarbon parameters are not known. Results for the Reference Samples are summarized in Table 1.

The results obtained for the seven method QC samples indicate that the QC acceptance criteria specified in the methods are reasonable and can be met by the majority of laboratories without undue effort or difficulty.

The BCMELP Hydrocarbon Methods

The Draft 2.0 hydrocarbon methods incorporate stakeholder input received following public release for comment of the Draft 1.0 methods in July, 1998.

Four analytical test methods were evaluated within this study:

- **Volatile Hydrocarbons (VH) in Solids**
- **Volatile Hydrocarbons (VH) in Water**
- **Extractable Petroleum Hydrocarbons (EPH) in Solids**
- **Extractable Petroleum Hydrocarbons (EPH) in Water**

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The new BC Hydrocarbon Methods were designed as Performance Based Methods. However, because the VH and EPH parameters are “method-defined”, critical elements within each method have been prescribed and are mandatory. This is necessary to ensure reasonable inter-laboratory comparability. Laboratories will be permitted to modify non-critical elements of the methods if they are able to verify and document that the modified procedure can provide results equivalent to that of the reference method. Requirements for demonstrating equivalence of alternate procedures are described within each method.

Round Robin Objectives

The primary objective of the round robin study was to evaluate the inter-laboratory variability of the draft hydrocarbon methods. The study was not intended to assess the proficiency of participating laboratories, nor was it intended to demonstrate the equivalence of any alternate or modified procedures. Therefore, ***all participating laboratories were asked to follow the methods as written, as closely as possible, with no significant modifications.*** In several instances, participating laboratories submitted results generated from modified procedures. Data generated by procedures that did not follow one or more prescribed elements of a particular method were excluded from statistical evaluation. However, these data are reported in Appendix A.

In addition to the four analytical test methods, two new calculation procedures were also evaluated:

- **Calculation of Volatile Petroleum Hydrocarbons (VPH) in Solids or Water**
- **Calculation of Light and Heavy Extractable Petroleum Hydrocarbons (LEPH & HEPH) in Solids or Water**

The parameters VPH, LEPH, and HEPH are scheduled “substances” under the Contaminated Sites Regulation. They are calculated parameters, determined by subtracting specified target parameter results from VH or EPH results. To allow evaluation of the VPH and LEPH/HEPH calculation procedures, the round robin study included the analysis of Mononuclear Aromatic Hydrocarbons (MAH) for all VH samples, and included the analysis of Polynuclear Aromatic Hydrocarbons (PAH) for all EPH samples.

The methods used to determine MAH and PAH are not directly evaluated in this study. However, their analysis is nevertheless an integral component of the new methodologies. Therefore, participating laboratories were asked to provide information about the methods used to determine these parameters.

Round Robin Organization and Administration

Environment Canada’s Pacific Environmental Science Center (PESC) was contracted by BCMELP to coordinate and administer the round robin. ASL Analytical Service Laboratories (ASL), the author of the new hydrocarbon methods, was contracted to design the study, to source and prepare all round robin sample materials, to prepare instructions to participants, and to complete this report.

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In late November 1998, round robin study materials were distributed by PESC to laboratories that had agreed to participate in the study. Of the twenty-two SCC/CAEAL accredited and certified laboratories in BC and Alberta¹, ten laboratories requested and received round robin materials. A total of nine laboratories ultimately contributed results to the round robin. The names of these participating laboratories are listed below.

PARTICIPATING LABORATORIES:

AGAT Laboratories	Calgary, AB
AGRA Earth & Environmental Limited	Edmonton, AB
ASL Analytical Service Laboratories Ltd.	Vancouver, BC
Can Test Ltd.	Vancouver, BC
Enviro-Test Laboratories	Edmonton, AB
Levelton Analytical Service	Richmond, BC
Maxxam Analytic Inc.	Calgary, AB
Norwest Labs	Surrey, BC
Philip Analytical Services Corporation	Burnaby, BC

Each participant was provided with a distribution package that included the following:

- A copy of the BCMELP Petroleum Hydrocarbon methods (Draft 2.0, October 98).
- Round robin study samples and spike solutions.
- Detailed instructions to participants.

By mid January 1999, PESC had received results from participating laboratories in both electronic and hard copy format. Several laboratories used procedures that contravened prescribed (i.e. required) elements of one or more of the four methods, and those results could not be used within this study to directly assess the methods. One laboratory did not have the capability to perform purge & trap analyses, and therefore did not submit data for VH/MAH in water.

ASL and PESC jointly reviewed the submitted data to identify transcription errors and calculation errors². Results generated using significantly modified methods were excluded from statistical evaluation. Where potential errors were identified, PESC contacted the laboratories to confirm results. For confirmed errors, corrected results were used for the study. Results that failed critical Quality Control (QC) requirements of the methods were also excluded from statistical evaluation. All data exclusions are flagged and noted in Appendix A.

Single Laboratory Results

In conjunction with the BCMELP Petroleum Hydrocarbon Round Robin, ASL Analytical Service Laboratories carried out single laboratory validation of the methods using the same Reference Samples and QC Samples as were used in the round robin. To assess intra-laboratory variability,

¹ About half of the 22 labs routinely analyze Petroleum Hydrocarbons for direct or indirect submission to BCMELP.

² Laboratories are identified anonymously by number. Laboratory identities are known only to PESC.

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ASL performed each analysis with at least 6 replicates (typically 8-9). Single laboratory results also include Method Detection Limit (MDL) determination data.

Summary of Round Robin and Single Laboratory Results

Table 1 summarizes and compares the key results from the round robin and single laboratory studies. Note that since the “true values” for any of the hydrocarbon round robin samples are unknown, only method variability and comparability can be assessed.

Table 1: COMPARISON OF ROUND ROBIN AND SINGLE LABORATORY RESULTS									
	Units	Round Robin		Single Lab		Round Robin		Single Lab	
		Mean	%RSD	Mean	%RSD	Mean	%RSD	Mean	%RSD
EPH in Water		Low Level Method Spike				High Level Method Spike			
EPH _{W10-19}	ug/L	3147	29.5%	3384	8.4%	16325	18.4%	17428	4.7%
EPH _{W19-32}	ug/L	579	16.9%	565	8.3%	2978	17.2%	3341	4.8%
Total PAH	ug/L	4.60	62.0%	3.51	6.8%	23.1	48.6%	21.6	5.7%
EPH in Solid		RTC Reference Material 355-100 (EPHRM1)				NRC Reference Material HS3B (EPHRM2)			
EPH _{S10-19}	mg/kg	3312	9.9%	3429	2.6%	385	18.0%	458	7.2%
EPH _{S10-19}	mg/kg	5038	17.7%	5284	1.9%	2745	26.6%	2456	4.0%
Total PAHs	mg/kg	10.3	34.7%	12.31	6.9%	52.7	36.2%	64.8	4.2%
VPH in Water		Low Level Method Spike				High Level Method Spike			
VH _{W6-oXylene}	ug/L	892	27.3%	864	5.0%	4688	29.2%	4259	2.2%
VH _{W6-oXylene-10}	ug/L	117	42.9%	169	4.7%	725	39.9%	1072	1.0%
VH _{W6-10}	ug/L	1010	25.6%	1033	4.7%	5412	25.4%	5330	1.8%
Total BTEX	ug/L	227	21.0%	229	1.9%	1098	23.3%	1135	1.9%
VPH in Solids³		Low Level Method Spike				High Level Method Spike			
VH _{S6-oXylene}	mg/kg	95.7	22.6%	111	5.5%	449	10.5%	554	4.5%
VH _{S6-oXylene-10}	mg/kg	15.5	15.3%	18.6	8.4%	88.9	7.1%	100	4.6%
VH _{S6-10}	mg/kg	111	21.5%	129	5.6%	538	9.1%	654	4.2%
Total BTEX	mg/kg	28.2	27.0%	36.0	2.8%	118	29.5%	186	1.7%

Table 1 shows that the means of the round robin study results agree very well with those of the single laboratory study. In most cases these means are within 10-20% of each other. This degree of consistency is very good considering the small sample size of much of the round robin data.

As expected, single laboratory results were considerably more precise than round robin results. Single laboratory precision for the hydrocarbon parameters averaged 4-5% relative standard deviation (RSD). Inter-laboratory precision for the same parameters averaged 20-25% RSD. It is

³ VPH in Solids represents direct injection summary data only.

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particularly noteworthy that the inter-laboratory precision of the hydrocarbon parameters is typically better than that seen for the target MAH and PAH compounds. This was an encouraging (and unexpected) finding. Historically, petroleum hydrocarbon methods have exhibited unacceptable inter-laboratory variability and inconsistency. The need to reduce such variability was the primary reason behind the development of BC Environment's new hydrocarbon methods.

It is also interesting to note that the inter-laboratory precision achieved in the current study was comparable to or better than the precision achieved in a similar round robin study of twenty-seven laboratories carried out by the Commonwealth of Massachusetts, Department of Environmental Protection in the Fall of 1997⁴.

DESIGN AND EXECUTION OF ROUND ROBIN STUDY

Several factors complicated the selection of appropriate samples for this study. First, no reference materials are currently certified for the VH or EPH parameters. Furthermore, because the parameters are method-defined, the "true values" for VH and EPH can only be estimated, even for samples spiked with known amounts of petroleum products.

Where feasible, commercially available reference materials were chosen for round robin solids samples, due to their homogeneity, proven stability, and ease of acquisition. For this study, two soil/sediment reference materials that contained EPH-range petroleum hydrocarbons were selected. The first, HS3B, is a marine harbour sediment material prepared by the National Research Council of Canada (Halifax). The second, CRM 355-100, is a soil which was fortified with diesel/motor oil specifically for this study by the Resource Technology Corporation of Wyoming (RTC).

Suitable reference materials are not available for VH-range hydrocarbons in solids. Consequently, for this study, gasoline spikes into a wetted clean sand matrix were used as the VH solids Reference Samples.

Aqueous spike samples of diesel and gasoline, respectively, were selected for the EPH and VH water Reference Samples. To avoid problems with sub-sampling and with degradation during transport and storage, participating laboratories were instructed to prepare their own aqueous Reference Samples using reagent water and the spike solutions provided.

Participants were also provided with mixed solutions of specific alkane and aromatic hydrocarbons, for use in the methods' Instrument Performance and Method Performance Checks. Instrument Performance Checks are used to measure and control the relative instrument responses of various VH and EPH components. Method Performance Checks are used to monitor and control losses of analyte throughout each procedure. Each participating laboratory was responsible for supplying and preparing its own instrument calibration standards. This procedure likely contributed to the variability of the inter-laboratory results. Participating laboratories were blind to the identities of

⁴Commonwealth of Massachusetts, Executive Office of Environmental Affairs, Department of Environmental Protection, "Report on Results of the Fall 1997 VPH/EPH Round Robin Testing Program", January 12, 1998.

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the reference materials, and to the types and concentrations of petroleum product spiking solutions used.

The study was not designed to evaluate performance of MAH and PAH methodologies, but these parameters form an integral component of the VPH and LEPH/HEPH procedures. Consequently, participating laboratories were asked to report MAH and PAH results.

Table 2 summarizes instructions provided to participants.

Table 2: SUMMARY OF ROUND ROBIN INSTRUCTIONS				
Method	Section	Type⁵	Sample	Instructions
EPHw	1.1	IP	Solution 1	Dilute Solution 1 50 times in iso-octane. Analyze directly according to method.
	1.2	MS	Solution 1	Spike 20 ul of Solution 1 into 500 mL of reagent water. Analyze according to method.
	1.3	MS	Solution 2	Spike 50 ul of Solution 2 into 500 mL of reagent water. Analyze according to method.
	1.4	MS	Solution 2	Spike 250 ul of Solution 2 into 500 mL of reagent water. Analyze according to method.
EPHs	2.1	IP	Solution 1	Dilute Solution 1 50 times in iso-octane. Analyze directly according to method.
	2.2	MS	Solution 1	Spike 100 ul of Solution 1 directly into extraction vessel containing 10 grams of spike matrix and 2 mL reagent water. Analyze according to method.
	2.3	RM	EPHRM1	Weigh 10 grams of EPHRM1 into extraction vessel, add 2 mL reagent water. Analyze according to method.
	2.4	RM	EPHRM2	Weigh 10 grams of EPHRM2 into extraction vessel, add 2 mL reagent water. Analyze according to method.
VHw	3.1	IP	Solution 3	Dilute Solution 3 50 times in methanol. Spike 1 ul of solution for every 1 mL of purge water. Spike directly into purge vessel. Analyze according to method.
	3.2	MS	Solution 4	Spike 1ul of Solution 4 for every 1 mL of purge water . Spike directly into purge vessel. Analyze according to method.
	3.3	MS	Solution 4	Spike 5ul of Solution 4 for every 1 mL of purge water. Spike directly into purge vessel. Analyze according to method.
VHs	4.1	IP	Solution 3	Dilute Solution 3 100 times in methanol. Analyze directly according to method.
	4.2	MS	Solution 3	Spike 220 ul of Solution 3 into 10 grams of spike matrix and 2 mL reagent water. Analyze according to method.
	4.3	MS	Solution 5	Spike 50 ul of Solution 5 into 10 grams of spike matrix and 2 mL reagent water. Analyze according to method.
	4.4	MS	Solution 5	Spike 250 ul of Solution 5 into 10 grams of spike matrix and 2 mL reagent water. Analyze according to method.

⁵ Sample Types: IP = instrument performance, MS = matrix spike, and RM = reference material.

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Table 3 lists the sources and descriptions of the round robin sample materials.

TABLE 3: DESCRIPTION OF ROUND ROBIN STUDY MATERIALS			
Sample	Manufacturer / Supplier	Catalog #	Description
Solution 1	Crescent Chemical Company Inc., Hauppauge, NY some solutions were custom-made	CCS-115R2	5,000 ug/mL of each of nC10, nC12, nC16, nC19, nC20, nC30, nC32, nC40, Naphthalene, Phenanthrene, Pyrene, and Benzo(a)pyrene in 1:1 Dichloromethane:Carbon Disulfide
Solution 2		PDR-002A.500	50,000 ug/mL diesel in acetone
Solution 3		CCS-119R	5,000 ug/mL of each of nC6, nC8, nC10, Benzene, Ethylbenzene, Toluene, m-Xylene, p-Xylene, and o- Xylene in methanol.
Solution 4		PGR-001M.20	2,000 ug/mL gasoline in methanol.
Solution 5		PGR-001M.500	50,000 ug/mL gasoline in methanol
EPHRM1	Resource Technology Corporation, Laramie, WY	CRM 355-100	Reference Material, soil matrix fortified with diesel and motor oil. Not certified.
EPHRM2	National Research Council Canada, Halifax, NS	HS3B	Reference Material, marine harbour sediment (certified for PAH).

DISCUSSION OF ROUND ROBIN AND SINGLE LABORATORY RESULTS

Summary results for the nine participating British Columbia and Alberta laboratories submitting results for this study are presented in Tables 4 through 18.

Since the intent of the study was to evaluate inter-laboratory variability of the hydrocarbon methods as written, laboratories were asked to use the methods without modification wherever possible. Where laboratories used significantly modified methodologies, their results were excluded for calculation of mean and percent relative standard deviation data. Refer to Appendix A for the complete tabulation of round robin results, together with pertinent information on the methods used by each laboratory. Scientific justification for exclusion of data is also presented in Appendix A.

Of the 9 laboratories that submitted data for EPH in Waters:

- All 9 labs followed the EPHw method with little or no modification.

Of the 9 laboratories that submitted data for EPH in Solids:

- 7 labs followed the EPHs method with little or no modification.
- 1 lab used Dichloromethane as the extraction solvent.
- 1 lab used a different method of extraction and a different ratio of hexane and acetone.

Of the 8 laboratories that submitted data for VH in waters:

- 7 labs followed the VHw method with little or no modification.
- 1 lab used headspace as the method of sample introduction.

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Of the 9 laboratories that submitted data for VH in solids:

- 6 labs followed the VHs method with little or no modification.
- 3 labs used purge & trap as the method of sample introduction.

The single laboratory data produced by ASL is discussed below in comparison with the round robin results. For VH in Solids, the single lab data also includes an additional matrix spike, representing a higher concentration than either of the round robin VHs spikes. Single laboratory Method Detection Limit (MDL) results are reported in Table 19. A complete tabulation of the single laboratory results is provided in Appendix B. For comparison purposes, summary results for the single laboratory data are presented with corresponding round robin summary results in Tables 4 through 18.

EPH in Water - Instrument Performance Check

Table 4: EPHw INSTRUMENT PERFORMANCE CHECK (Part 1.1)						
Relative Response	Round Robin Results			Single Lab Results		
	(n)	Mean	% RSD	(n)	Mean	% RSD
Decane (nC10)	6	0.99	8.3%	8	0.99	0.8%
Naphthalene	6	1.03	7.0%	8	1.02	1.0%
Dodecane (nC12)	4	0.97	2.8%	8	1.01	1.6%
Hexadecane (nC16)	7	0.99	2.8%	8	0.99	0.8%
Phenanthrene	7	1.04	5.7%	8	1.00	1.3%
Nonadecane (nC19)	7	1.00	0.8%	8	0.99	0.7%
Eicosane (nC20)	7	1.00	n/a	8	1.00	n/a
Pyrene	7	1.08	1.5%	8	1.01	3.6%
Benzo(a)pyrene	6	0.82	19.6%	-	-	-
Triacontane (nC30)	6	0.92	15.5%	8	0.98	5.6%
Dotriacontane (nC32)	7	0.87	16.0%	8	0.92	8.4%

EPHw is analyzed by direct injection Gas Chromatography with Flame Ionization Detection (GC-FID). The EPHw Instrument Performance Check is a required QC component of the EPHw method. It is designed to ensure that the GC-FID instrument is operating such that the response of hydrocarbon components throughout the EPH range are approximately equal, thus preventing relative bias between higher and lower molecular weight EPH components, and making inter-laboratory consistency possible.

Instrument Performance Checks have been incorporated into the new BC Environment hydrocarbon methods. They are the primary measure of instrument performance for the methods, and they govern which modifications to *instrumental components* of the new methods are permissible under the performance based methods approach.

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Results for the EPHw Instrument Performance Check are reported as “relative response”, which is the GC-FID peak area of each specified component divided by the peak area of eicosane (nC20), where all components are present at the same concentration. A relative response of 1.00 indicates equal response between the specified component and nC20. The EPHw method states that the relative response versus nC20 of all compounds listed in Table 4 must fall between 0.7 – 1.3 (except in the case of benzo(a)pyrene, for which monitoring is not required).

Table 4 summarizes the results from the seven laboratories that passed (or almost passed) the method acceptance criteria for this sample.

Data from two laboratories were excluded due to their extremely low relative responses in the nC30 to nC32 range. Results from those two laboratories were also excluded from statistical evaluations of other EPHw round robin samples.

For both the round robin and single laboratory results, the mean relative response of all compounds in this sample (except benzo(a)pyrene) ranged from approximately 0.9 – 1.0. This result indicates that the instrumental procedure specified in the EPHw method provides equivalent response throughout the EPH range of nC19 through nC32.

EPH in Water - Method Performance Check

Table 5: EPHw METHOD PERFORMANCE CHECK (Part 1.2)						
	Round Robin Results			Single Lab Results		
Spike Recovery (%)	(n)	Mean	% RSD	(n)	Mean	% RSD
Decane (nC10)	6	54.9%	18.6%	8	85.6%	4.5%
Naphthalene	6	82.7%	31.3%	8	101%	5.2%
Dodecane (nC12)	4	69.3%	15.3%	8	99.0%	5.0%
Hexadecane (nC16)	7	92.1%	18.1%	8	105%	5.5%
Phenanthrene	7	93.1%	17.1%	8	105%	6.1%
Nonadecane (nC19)	7	94.7%	17.9%	8	105%	5.6%
Eicosane (nC20)	7	95.4%	17.0%	8	105%	5.6%
Pyrene	7	93.9%	15.0%	8	106%	7.1%
Benzo(a)pyrene	6	99.9%	19.9%	8	111%	6.0%
Triacontane (nC30)	6	94.8%	19.3%	8	110%	6.1%
Dotriacontane (nC32)	7	97.8%	20.4%	8	102%	5.5%

The EPHw Method Performance Check is a required QC component of the EPHw method. It is designed to monitor potential losses of EPH components through the sample preparation steps of the method. It governs which modifications to *sample preparation components* of the new methods are permissible under the performance based methods approach.

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Results are reported as “spike recovery”, which is the measured concentration divided by the spiked concentration of each compound (in percent). The EPHw method states that the recovery of all compounds listed in Table 5 must normally range between 80% and 120% for nC13 through nC32, and between 65% and 120% for nC10, nC12, and naphthalene.

For both the round robin and single laboratory results, the mean recoveries of most compounds ranged between 90-110%. This result indicates that the sample preparation procedures specified in the EPHw method can achieve reliable and consistent recoveries of compounds across the EPH range.

Round robin mean recoveries were slightly low for nC10 (55%) and for nC12 (69%). These results indicate loss due to volatility. These losses are most likely due to minor difficulties with spiking techniques (see results for EPHs method performance spikes). However, they may also indicate losses of volatiles during extract concentration steps or during the extraction process. Recoveries for these compounds were much better within the single laboratory data.

EPH in Water - Method Spikes

Table 6: EPHw LOW LEVEL METHOD SPIKE (Part 1.3)						
	Round Robin Results			Single Lab Results		
EPH Results (ug/L)	(n)	Mean	% RSD	(n)	Mean	% RSD
EPH _{W10-19}	7	3147	29.5%	8	3384	8.4%
EPH _{W19-32}	7	579	16.9%	8	565	8.3%
LEPHw	7	3143	29.5%	8	3380	8.4%
HEPHw	7	579	16.9%	8	565	8.3%
PAH Results (ug/L)	(n)	Mean	% RSD	(n)	Mean	% RSD
Naphthalene	8	1.72	40.7%	8	0.91	13.1%
Acenaphthene	5	0.49	67.1%	8	<0.5	n/a
Fluorene	8	0.78	53.8%	8	0.71	3.8%
Phenanthrene	8	1.30	45.0%	8	1.52	3.0%
Anthracene	6	<	n/a	8	0.21	7.2%
Acridine	5	<	n/a	8	<0.1	n/a
Fluoranthene	6	<	n/a	8	<0.05	n/a
Pyrene	6	0.20	62.8%	8	0.16	6.8%
Benz(a)anthracene	6	<	n/a	8	<0.05	n/a
Benzo(a)pyrene	7	<	n/a	8	<0.05	n/a

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Table 7: EPH_w HIGH LEVEL METHOD SPIKE (Part 1.4)						
Round Robin Results				Single Lab Results		
EPH Results (ug/L)	(n)	Mean	% RSD	(n)	Mean	% RSD
EPH _{W10-19}	7	16325	18.4%	8	17428	4.7%
EPH _{W19-32}	7	2978	17.2%	8	3341	4.8%
LEPH _w	7	16304	18.4%	8	17407	4.7%
HEPH _w	7	2978	17.2%	8	3340	4.8%
PAH Results (ug/L)	(n)	Mean	% RSD	(n)	Mean	% RSD
Naphthalene	8	9.56	38.4%	8	8.12	19.9%
Acenaphthene	6	2.62	59.1%	8	<3	n/a
Fluorene	8	3.73	47.4%	8	3.99	1.2%
Phenanthrene	8	6.36	43.4%	8	7.74	1.8%
Anthracene	4	<	n/a	8	0.98	3.4%
Acridine	6	<	n/a	8	<0.5	n/a
Fluoranthene	7	<	n/a	8	<0.2	n/a
Pyrene	6	0.85	54.8%	8	0.76	2.3%
Benz(a)anthracene	7	<	n/a	8	<0.05	n/a
Benzo(a)pyrene	7	<	n/a	8	<0.05	n/a

Tables 6 and 7 summarize round robin and single laboratory results for two method spikes of diesel into reagent water. Comparative equivalency between single laboratory and round robin data is excellent. Round robin precision for EPH_{W10-19} and EPH_{W19-32} was two to three times better than that seen for the PAH parameters.

The diesel concentrations of the spikes were 5,000 and 25,000 ug/L in water. There is no known "true value" for EPH_w in diesel spikes. For the 5,000 ug/L diesel spike, total EPH (i.e. the sum of EPH_{W10-19}+EPH_{W19-32}) for round robin and single laboratory data averaged 3,726 and 3,949 ug/L respectively. These results indicate that approximately 20-25% of the components of this diesel product lie outside the nC10-nC32 range, assuming no losses during sample processing steps.

The maximum reporting detection limit (DL) specified for each of EPH_{W10-19} and EPH_{W19-32} in the LEPH/HEPH Calculation Procedure is 250 ug/L. For EPH_{W10-19} in the low spike, mean results were approximately twelve times this level. For EPH_{W19-32}, mean results were two times the maximum reporting DL.

Method Detection Limits determined for single laboratory data were 59 ug/L for EPH_{W10-19} and 102 ug/L for EPH_{W19-32}.

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EPH in Solids - Instrument Performance Check

Table 8: EPHs INSTRUMENT PERFORMANCE CHECK (Part 2.1)						
Relative Response	Round Robin Results			Single Lab Results		
	(n)	Mean	% RSD	(n)	Mean	% RSD
Decane (nC10)	6	0.98	6.3%	8	1.01	1.8%
Naphthalene	6	1.03	6.9%	8	1.07	1.3%
Dodecane (nC12)	4	0.98	3.1%	8	1.00	1.4%
Hexadecane (nC16)	7	0.99	2.8%	8	1.00	1.5%
Phenanthrene	7	1.05	4.6%	8	1.06	0.8%
Nonadecane (nC19)	7	1.00	0.8%	8	0.99	0.4%
Eicosane (nC20)	7	1.00	n/a	8	1.00	n/a
Pyrene	7	1.07	3.3%	8	1.08	1.3%
Benzo(a)pyrene	6	0.87	13.6%	8	0.92	1.8%
Triacontane (nC30)	5	0.90	17.2%	8	1.02	1.5%
Dotriacontane (nC32)	7	0.90	16.1%	8	1.00	1.3%

EPHs is analyzed by direct injection Gas Chromatography with Flame Ionization Detection (GC-FID). The EPHs Instrument Performance Check is a required QC component of the EPHs method. It is designed to ensure that the instrument is operating such that the response of hydrocarbon components throughout the EPH range are approximately equal, thus preventing relative bias between higher and lower molecular weight EPH components, and making good inter-laboratory consistency possible.

The Instrument Performance Check is the primary measure of EPHs instrument performance. It governs which modifications to *instrumental components* of the new methods are permissible under the performance based methods approach.

This sample is identical to that of the EPHw Instrument Performance Check in Table 4.

Results for the EPHs Instrument Performance Check are reported as “relative response”, which is the GC-FID peak area of each specified component divided by the peak area of eicosane (nC20), where all components are present at the same concentration. A relative response of 1.00 indicates equal response between the specified component and nC20. The EPHs method states that the relative response versus nC20 of all compounds listed in Table 8 must fall between 0.7 – 1.3 (except in the case of benzo(a)pyrene, for which monitoring is not required).

Table 8 summarizes the results from the seven laboratories that passed (or almost passed) the method acceptance criteria for this sample.

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Data from two laboratories were excluded due to their extremely low relative responses in the nC30 through nC32 range. Results from those two laboratories were also excluded from statistical evaluations of other EPHs round robin samples.

For both the round robin and single laboratory results, the mean relative response of all compounds (except benzo(a)pyrene) in this sample ranged from approximately 0.9 – 1.1. This result indicates that the instrumental procedure specified in the EPHs method provides equivalent response throughout the EPH range of nC19 through nC32.

EPH in Solids - Method Performance Check

Table 9: EPHs METHOD PERFORMANCE CHECK (Part 2.2)						
	Round Robin Results			Single Lab Results		
Spike Recovery (%)	(n)	Mean	% RSD	(n)	Mean	% RSD
Decane (nC10)	4	41.6%	89.0%	8	89.1%	4.6%
Naphthalene	4	43.6%	52.2%	8	92.0%	3.5%
Dodecane (nC12)	3	40.9%	60.7%	8	92.0%	3.6%
Hexadecane (nC16)	6	80.9%	15.2%	8	95.4%	2.8%
Phenanthrene	6	75.1%	25.8%	8	94.9%	3.5%
Nonadecane (nC19)	6	83.6%	20.1%	8	96.5%	1.7%
Eicosane (nC20)	6	83.4%	23.7%	8	97.0%	1.5%
Pyrene	6	68.2%	41.8%	8	96.3%	1.7%
Benzo(a)pyrene	3	68.2%	45.0%	8	73.6%	6.5%
Triacontane (nC30)	3	88.0%	8.4%	8	97.0%	1.7%
Dotriacontane (nC32)	5	94.7%	17.9%	8	96.6%	1.7%

The EPHs Method Performance Check is a required QC component of the EPHs method. It is designed to monitor potential losses of EPH constituents through the sample preparation steps of the method. It governs which modifications to *sample preparation components* of the new methods are permissible under the performance based methods approach.

Results are reported as “spike recovery”, which is the measured concentration divided by the spiked concentration of each compound (in percent). The EPHs method states that the recovery of all compounds listed in Table 9 must normally range between 80% and 120% for nC13 through nC32, and between 65% and 120% for nC10, nC12, and naphthalene.

Round robin results for this sample were highly variable, due to a problem with the spiking technique that was not identified until after the study had begun. The round robin instructions specified the addition of 100uL of a spiking solution into 10 g of sand matrix plus 2 mL reagent water (Initial instructions specified 20uL of the spiking solution. However, this was changed to 100 uL in a letter of correction subsequently sent to participating laboratories). The sample was then to be dried with diatomaceous earth prior to Soxhlet extraction. Since the spiking solution was

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prepared in a highly volatile solvent mixture, and because such a small volume of the solution was added, evaporative losses during the drying step were severe.

Prior to the generation of the single laboratory data, the spiking problem was corrected by preparing the spike solution in iso-octane, a much less volatile solvent, and by adding a larger volume of the spike solution (1 mL) to Method Performance Check samples. Iso-octane simulates the presence of organic matter which is otherwise absent in a clean sand matrix (but which is normally present in soils and most sediments). If the spike matrix contains no organic matter, spiked volatile organics are lost much more quickly to evaporation. The corrected spiking procedure will be included the finalized version of the EPHs method.

Single laboratory data generated using the modified spiking technique achieved mean recoveries ranging between 90-100% for all analytes except benzo(a)pyrene, which had a mean recovery of 74%. This result indicates that the sample preparation procedures specified in the corrected EPHs method can yield reliable and consistent recoveries of compounds across the EPH range.

EPH in Solids - Analysis of Reference Materials

Table 10: EPHs REFERENCE MATERIAL 1 - RTC TPH 355-100 (Part 2.3)						
Round Robin Results				Single Lab Results		
EPH Results (mg/kg)	(n)	Mean	% RSD	(n)	Mean	% RSD
EPH _{S10-19}	6	3312	9.9%	8	3429	2.6%
EPH _{S19-32}	6	5038	17.7%	8	5284	1.9%
LEPHs	6	3302	9.9%	8	3417	2.6%
HEPHs	6	5038	17.7%	8	5283	1.9%
PAH Results (mg/kg)	(n)	Mean	% RSD	(n)	Mean	% RSD
Naphthalene	8	4.06	28.9%	8	4.47	4.9%
Phenanthrene	8	5.34	34.8%	8	6.87	4.5%
Pyrene	8	0.69	50.3%	8	0.75	1.9%
Benz(a)anthracene	4	0.11	55.5%	8	0.08	3.0%
Benzo(b)fluoranthene	3	0.04	20.8%	8	0.05	6.7%
Benzo(k)fluoranthene	3	0.02	75.8%	8	0.01	8.9%
Benzo(a)pyrene	3	0.05	32.8%	8	0.05	4.7%
Indeno(1,2,3-cd)pyrene	3	0.02	2.8%	8	0.02	3.0%
Dibenz(a,h)anthracene	3	0.01	10.8%	8	0.01	24.5%

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Table 11: EPHs REFERENCE MATERIAL 2 - NRC Canada HS3B (Part 2.4)						
	Round Robin Results			Single Lab Results		
EPH Results (mg/kg)	(n)	Mean	% RSD	(n)	Mean	% RSD
EPH _{S10-19}	5	385	18.0%	11	458	7.2%
EPH _{S19-32}	5	2745	26.6%	11	2456	4.0%
LEPHs	5	369	17.6%	11	439	7.2%
HEPHs	5	2707	26.6%	11	2411	4.0%
PAH Results (mg/kg)	(n)	Mean	% RSD	(n)	Mean	% RSD
Naphthalene	8	1.62	31.1%	11	1.82	5.2%
Phenanthrene	8	14.91	27.4%	11	17.56	6.1%
Pyrene	8	13.72	28.9%	11	15.75	2.1%
Benz(a)anthracene	8	5.85	35.6%	11	7.07	4.7%
Benzo(b)fluoranthene	8	6.50	43.5%	11	8.90	2.9%
Benzo(k)floranthene	8	3.25	41.1%	11	3.40	3.3%
Benzo(a)pyrene	8	3.79	28.8%	11	5.23	3.7%
Indeno(1,2,3-cd)pyrene	7	2.51	39.6%	11	3.99	2.5%
Dibenz(a,h)anthracene	7	0.57	50.2%	11	1.08	7.4%

Tables 10 and 11 present summarized round robin and single laboratory results for two commercially available sediment/soil reference materials (RMs). Each RM was wetted prior to extraction. Comparative equivalency between the single laboratory and round robin data is excellent. Round robin precision for EPH_{S10-19} and EPH_{S19-32} is consistently better than that seen for PAH parameters.

Reference Material 1 (RTC CRM-355) is a soil fortified with a diesel / motor oil mixture. Resource Technology Corporation (Laramie, Wyoming) produced this RM specifically for this study. This RM has not been dried, consequently it contains a significant portion of lighter EPH constituents.

Reference Material 2 (NRC HS3B) is a marine harbour sediment which has been dried and sieved. It is produced by the National Research Council of Canada (Halifax), and is certified for PAH content. EPH content of this RM is primarily in the nC19-nC32 range.

Results from two laboratories were excluded due to the use of significantly modified methods. One lab used a DCM Soxhlet extraction, and the other used DCM shaker extraction. The method requires the use of 1:1 hexane:acetone as the extraction solvent.

As with the EPHw diesel spikes, there is no “true value” for EPHs in either of these materials.

Round robin and single laboratory precision was better for reference material 1 (i.e. the RTC RM) than for reference material 2 (i.e. the NRC RM). This is likely due to the higher concentrations

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inherent in reference material 1. However, it is also possible that reference material 1 may be easier to extract, since it is a fortified material.

The maximum reporting detection limit (DL) specified for EPH_{S10-19} and EPH_{S19-32} in the LEPH/HEPH Calculation Procedure is 500 mg/kg. Reference material 2 (i.e. the NRC RM) contains EPH_{S10-19} at a concentration approximately equal to 500 mg/kg, and contains EPH_{S19-32} at about five times this level. Reference material 1 (i.e. the RTC RM) contains both EPH parameters at approximately 7-10 times the maximum reporting DL.

Method Detection Limits determined for the single laboratory data were 21 mg/kg for EPH_{S10-19} and 9 mg/kg for EPH_{S19-32}.

VH in Water - Instrument and Method Performance Check

Table 12: VHw INSTRUMENT / METHOD PERFORMANCE CHECK (Part 3.1)						
	Round Robin Results			Single Lab Results		
Relative Response	(n)	Mean	% RSD	(n)	Mean	% RSD
Hexane (nC6) ⁶	5	0.96	25.8%	7	0.98	0.3%
Benzene ⁶	5	1.09	9.4%	7	1.09	0.1%
Toluene ⁶	5	1.04	4.9%	7	1.05	0.1%
Octane (nC8) ⁶	5	0.90	25.1%	7	0.98	0.0%
Ethylbenzene ⁶	5	0.99	4.1%	7	1.01	0.0%
m,p-Xylene ⁶	5	1.00	n/a	7	1.00	n/a
o-Xylene ⁷	4	1.08	6.4%	7	0.96	0.0%
1,2,4-Trimethylbenzene ⁷	5	1.00	n/a	7	1.00	n/a
Decane (nC10) ⁷	4	0.76	24.0%	7	0.86	0.3%

VHw is analyzed by Purge and Trap - Gas Chromatography with Flame Ionization Detection (P&T-GC-FID). The VHw Instrument / Method Performance Check is a required QC component of the VHw method. It is designed to ensure that the P&T-GC-FID instrument is operating such that the response of hydrocarbon components throughout the VH range are approximately equal, thus preventing relative bias between higher and lower molecular weight VH components, or between aliphatic and aromatic VH components. This step enhances inter-laboratory consistency.

The Instrument / Method Performance Check is the primary measure of VHw method performance. It governs which modifications to *instrumental and sample preparation components* of the new methods are permissible under the performance based methods approach.

⁶ Relative response calculated against m,p-Xylene.

⁷ Relative response calculated against 1,2,4-Trimethylbenzene.

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For VHw, the instrument and method performance checks are combined, because water samples analyzed by purge and trap require no additional sample extraction or sample processing steps. Thus, for VHw, an Instrument Performance Check is identical to a Method Performance Check.

Results for the VHw Instrument/Method Performance Check are reported as “relative response”, which is the GC-FID peak area of each specified component divided by the peak area of m,p-xylene or 1,2,4-trimethylbenzene, where all components are present at the same concentration. A relative response of 1.00 indicates equal response between the two compounds. The VHw method states that the relative response (versus m,p-xylene or 1,2,4-trimethylbenzene) of all compounds listed in Table 12 must fall between 0.6 – 1.4.

Table 12 summarizes the results from the five laboratories that followed the required analysis procedure, and that passed the method acceptance criteria for this sample.

Data from three laboratories were excluded due to poor relative responses of one or more components, or due to the failure to use purge and trap instrumentation. Results from these laboratories were also excluded from statistical evaluations of other VHw round robin samples. One of the three labs used the headspace technique, which is not permitted in the VHw method, since it is known to exacerbate relative bias between aliphatic and aromatic compounds. (Note the bias of up to +275% for aliphatics in the results presented for lab 8 in Appendix A, Part 3.1, VPHw Instrument and Method Performance Check).

For both the round robin and single laboratory results, the mean relative response of most compounds in this sample ranged from approximately 0.9 – 1.1. Mean relative response for decane was slightly lower at 0.76 – 0.86. These results indicate that the instrumental procedure specified in the VHw method can achieve equivalent response throughout most of the VH range, with a slight decline in the nC9-nC10 region.

Note that a decline in purge and trap instrument response in the nC9-nC10 region was the reason that the VH Draft 2.0 methods were modified to use a “two-range” calibration mode. The nC6 to ortho-xylene range is now calculated against meta-xylene, and the ortho-xylene to nC10 range is now calculated against 1,2,4-trimethylbenzene.

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VH in Water – Method Spikes

Table 13: VHw LOW LEVEL METHOD SPIKE (Section 3.2)

EPH _{W6-oXylene}		Round Robin Results			Single Lab Results		
EPH _{S19-32}							
VH Results(ug/L)	(n)	Mean	% RSD	(n)	Mean	% RSD	
VH _{W6-oXylene}	5	892	27.3%	8	864	5.0%	
VH _{WoXylene-10}	5	117	42.9%	8	169	4.7%	
VH _{W6-10}	5	1010	25.6%	8	1033	4.7%	
VPHw	4	880	26.7%	8	806	6.0%	
BTEX Result (ug/L)	(n)	Mean	% RSD	(n)	Mean	% RSD	
Benzene	6	17.1	13.5%	8	15.4	0.8%	
Toluene	6	90.0	19.6%	8	107	3.0%	
Octane (nC8)	6	21.4	18.7%	8	20.5	1.6%	
Ethylbenzene	6	72.9	37.6%	8	60.2	2.3%	
m,p-Xylene	6	25.4	15.7%	8	23.7	1.7%	
o-Xylene	4	1.08	6.4%	7	0.96	0.0%	

Table 14: VHw HIGH LEVEL METHOD SPIKE (Section 3.3)

		Round Robin Results			Single Lab Results		
VH Results(ug/L)	(n)	Mean	% RSD	(n)	Mean	% RSD	
VH _{W6-oXylene}	5	4688	29.2%	8	4259	2.2%	
VH _{WoXylene-10}	5	725	39.9%	8	1072	1.0%	
VH _{W6-10}	5	5412	25.4%	8	5330	1.8%	
VPHw	3	3817	16.1%	8	4195	2.1%	
BTEX Result (ug/L)	(n)	Mean	% RSD	(n)	Mean	% RSD	
Benzene	4	84.6	18.4%	8	76.8	0.8%	
Toluene	4	466	17.5%	8	536	3.0%	
Ethylbenzene	4	103	27.9%	8	102	1.6%	
m,p-Xylene	4	323	27.7%	8	301	2.3%	
o-Xylene	4	121	24.8%	8	119	1.7%	

Tables 13 and 14 present summarized round robin and single laboratory results for two method spikes of gasoline into reagent water. Comparative equivalency between single laboratory and round robin data is very good. Round robin precision for VH_{W6-10} was approximately equal to that for the MAH (i.e. BTEX) parameters.

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Gasoline concentrations of the spikes were 2,000 and 10,000 ug/L in water. There is no known “true value” for VHw in the gasoline spikes. For the 2,000 ug/L spike, VH_{W6-10} in round robin and single laboratory data average 1010 and 1033 ug/L respectively. This result indicates that approximately 50% of the composition of this gasoline product lies outside the nC6-nC10 range, assuming no losses during sample preparation steps.

The maximum reporting detection limit (DL) specified for VH_{W6-10} in the VPH Calculation Procedure is 100 ug/L. For VH_{W6-10} in the low spike, mean results were approximately ten times this level.

The Method Detection Limit for VH_{W6-10}, determined for the single laboratory data, was 13 ug/L.

For the analysis of BTEX, five labs used GC with Mass Spectrometric detection (GC/MS), which is the preferred instrument. One lab used GC with Photo Ionization Detection (GC/PID). The GC/MS and the GC/PID results exhibited reasonable consistency for these samples. Two labs used GC with Flame Ionization Detection (GC/FID). GC/FID results were less consistent than GC/MS or GC/PID results for these samples.

VH in Solids – Instrument Performance Check

Table 15: VPHs INSTRUMENT PERFORMANCE CHECK (Part 4.1)									
	Round Robin Results						Single Lab Results		
	Direct Injection			Purge & Trap			Direct Injection		
Relative Response	(n)	Mean	% RSD	(n)	Mean	% RSD	(n)	Mean	% RSD
Hexane (nC6) ⁸	4	0.78	9.0%	2	1.10	28.1%	9	0.81	0.7%
Benzene ⁸	5	1.00	4.9%	2	1.14	11.1%	9	1.01	0.5%
Toluene ⁸	5	0.99	3.5%	2	1.07	6.5%	9	1.00	0.2%
Octane (nC8) ⁸	5	0.91	2.7%	2	0.97	11.2%	9	0.91	0.4%
Ethylbenzene ⁸	5	0.99	3.1%	2	1.02	2.4%	9	0.99	0.0%
m,p-Xylene ⁸	5	1.00	n/a	2	1.00	n/a	9	1.00	n/a
o-Xylene ⁹	5	1.07	2.6%	2	1.12	5.5%	9	1.02	0.2%
1,2,4-Trimethylbenzene ⁹	5	1.00	n/a	2	1.00	n/a	9	1.00	n/a
Decane (nC10) ⁹	5	0.89	4.3%	2	0.78	10.0%	9	0.96	0.3%

VHs is analyzed by direct injection Gas Chromatography with Flame Ionization Detection (GC-FID). The VHs Instrument Performance Check is a required QC component of the VHs method. It is designed to ensure that the GC-FID instrument is operating such that the response of hydrocarbon components throughout the VH range are approximately equal, thus preventing relative bias

⁸ Relative response calculated against m,p-Xylene.

⁹ Relative response calculated against 1,2,4-Trimethylbenzene.

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between higher and lower molecular weight VH components, or between aliphatic and aromatic VH components. This step enhances inter-laboratory consistency.

The Instrument Performance Check is the primary measure of VHs instrument performance. It governs which modifications to *instrumental components* of the new methods are permissible under the performance based methods approach.

Results are reported as “relative response”, which is the GC-FID peak area of each specified component divided by the peak area of meta,para-xylene or 1,2,4-trimethylbenzene, where all components are present at the same concentration. A relative response of 1.00 indicates equal response between the two compounds. The VHs method states that the relative response (versus m,p-xylene or 1,2,4-trimethylbenzene) of all compounds listed in Table 15 must fall between 0.7 – 1.3.

Table 15 summarizes the results from the seven laboratories that passed the method acceptance criteria for this sample. Results for two different analytical techniques (i.e. direct injection and purge & trap) are summarized independently.

Data from two laboratories were excluded due to poor relative responses of one or more components.

Three laboratories used purge & trap instrumentation for the VHs round robin samples. Purge & trap is not the reference technique called for by this method. However, it is permitted as an alternative procedure under the performance based method approach (laboratories must first demonstrate equivalence to the reference procedure). Therefore, direct injection results and purge and trap results were summarized separately.

The direct injection results demonstrate the performance of the VHs method as written. The purge & trap summary results are provided for reference purposes only. However, comparison of the results for the two techniques does indicate that equivalence between them can probably be achieved. (Note the small sample population for the purge & trap method, with n=2).

For both single laboratory and round robin results by either direct injection or purge & trap, the mean relative response of most compounds in this sample ranged from approximately 0.9 – 1.1, with two exceptions:

- The direct injection procedure exhibited a slight low-bias for hexane, with a relative response was approximately 0.8 relative to m,p-xylene.
- The purge and trap procedure exhibited a slight low-bias for decane, with a relative response of approximately 0.8 relative to 1,2,4-trimethylbenzene.

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Both instrumental procedures are capable of generating equivalent response throughout most of the VH range, but direct injection exhibits slightly lower relative response in the nC6-nC7 region, and purge & trap shows exhibits slightly lower relative response in the nC9-nC10 region.

Note that the decreased response of purge and trap in the nC9-nC10 region was the reason that the VH Draft 2.0 methods were modified to use a “two-range” calibration mode. Without this change, the use of purge and trap bias would cause a much more significant negative bias in VH results. The nC6 to o-xylene range is now calculated against m-xylene, and the o-xylene to nC10 range is now calculated against 1,2,4-trimethylbenzene.

VH in Solids – Method Performance Check

Table 16: VPHs – METHOD PERFORMANCE CHECK (Part 4.2)									
	Round Robin Results						Single Lab Results		
	Direct Injection			Purge & Trap			Direct Injection		
Spike Recovery (%)	(n)	Mean	% RSD	(n)	Mean	% RSD	(n)	Mean	% RSD
Hexane (nC6)	4	102%	4.7%	2	97.3%	8.6%	8	88.0%	10.4%
Benzene	5	104%	4.1%	2	104%	5.3%	8	101%	7.0%
Toluene	5	105%	5.9%	2	102%	0.1%	8	100%	5.8%
Octane (nC8)	5	103%	4.8%	2	103%	3.1%	8	97.5%	8.1%
Ethylbenzene	5	106%	3.9%	2	100%	3.5%	8	100%	5.3%
m,p-Xylene	5	106%	4.0%	2	103%	0.2%	8	100%	5.4%
o-Xylene	5	106%	4.2%	2	102%	0.9%	8	101%	5.9%
1,2,4-Trimethylbenzene	5	109%	3.5%	2	105%	3.6%	8	103%	6.7%
Decane (nC10)	5	105%	4.9%	2	110%	4.8%	8	98.3%	7.2%

The VHs Method Performance Check is a required QC component of the VHs method. It is designed to monitor potential losses of VH range components through the sample preparation steps of the method. It governs which modifications to *sample preparation components* of the new methods are permissible under the performance based methods approach.

Results are reported as “spike recovery”, which is the measured concentration divided by the spiked concentration of each compound (in percent). The VHs method states that the recovery of all compounds listed in Table 16 must normally range between 80% and 120%.

The round robin and single laboratory results were highly comparable, exhibiting mean recoveries ranging from 90-110%. This indicates that the sample preparation procedures specified in the VHs method can generate reliable and consistent recoveries for compounds across the VH range. Round robin precision for this sample was excellent. (Note that in this case, round robin precision is actually better than for the single laboratory).

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VH in Solids – Method Spikes

Table 17: VPHs – LOW LEVEL METHOD SPIKE (Part 4.3)									
	Round Robin Results						Single Lab Results		
	Direct Injection			Purge & Trap			Direct Injection		
VH Results(mg/kg)	(n)	Mean	% RSD	(n)	Mean	% RSD	(n)	Mean	% RSD
VH _{S6-oXylene}	5	95.7	22.6%	2	147	44.2%	8	111	5.5%
VH _{SoXylene-10}	5	15.5	15.3%	2	15.3	17.9%	8	18.6	8.4%
VH _{S6-10}	5	111	21.5%	2	163	38.3%	8	129	5.6%
VPHs	4	87.3	26.0%	2	138	37.4%	8	93.3	7.6%
BTEX Result (mg/kg)	Combined Data						Direct Injection		
	(n)	Mean		% RSD		(n)	Mean	% RSD	
Benzene	7	2.2		25.9%		8	2.6	3.2%	
Toluene	7	11.2		31.5%		8	15.8	3.1%	
Ethylbenzene	7	2.7		26.8%		8	3.4	2.1%	
m,p-Xylene	7	8.9		26.4%		8	10.3	3.1%	
o-Xylene	6	3.3		24.1%		8	4.0	2.7%	
Styrene	9	<		n/a		8	<0.01	n/a	

Table 18: VPHs – HIGH LEVEL METHOD SPIKE (Part 4.4)									
	Round Robin Results						Single Lab Results		
	Direct Injection			Purge & Trap			Direct Injection		
VH Results(mg/kg)	(n)	Mean	% RSD	(n)	Mean	% RSD	(n)	Mean	% RSD
VH _{S6-oXylene}	5	449	10.5%	2	710	53.6%	6	554	4.5%
VH _{SoXylene-10}	5	88.9	7.1%	2	87.3	25.4%	6	100	4.6%
VH _{S6-10}	5	538	9.1%	2	797	50.4%	6	654	4.2%
VPHs	4	422	16.5%	2	699	51.7%	6	468	5.6%
BTEX Result (mg/kg)	Combined Data						Direct Injection		
	(n)	Mean		% RSD		(n)	Mean	% RSD	
Benzene	7	8.9		35.5%		6	12.6	1.6%	
Toluene	7	46.3		24.7%		6	81.0	1.3%	
Ethylbenzene	7	11.5		32.1%		6	17.9	2.0%	
m,p-Xylene	7	37.3		25.6%		6	53.4	2.2%	
o-Xylene	6	13.9		29.3%		6	20.9	1.4%	
Styrene	8	<		n/a		6	<0.05	n/a	

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Tables 17 and 18 present summarized round robin and single laboratory results for two method spikes of gasoline into a wetted clean sand matrix. Comparative equivalency between single laboratory and round robin direct injection data is good. Round robin precision for VH_{S6-10} by direct injection was approximately three times better than for the BTEX parameters. Round robin variability for VH_{S6-10} appeared to be higher by the purge & trap technique, but this measurement was based on only two samples, and so is unlikely to be representative (recall also that purge and trap is not the reference technique for this method).

The gasoline concentrations of the spikes were 250 and 1,250 mg/kg in clean sand. There is no “true value” for VHs in these gasoline spikes. For the 250 mg/kg spike, average direct injection round robin and single laboratory results for VH_{S6-10} were 111 and 129 mg/kg respectively. These results indicate that approximately 50% of the composition of this gasoline product lies outside the nC6-nC10 range, assuming no losses during sample processing steps.

The maximum reporting detection limit (DL) specified for VH_{S6-10} in the VPH Calculation Procedure is 100 mg/kg. For VH_{S6-10} in the low spike, mean results were approximately equal to this level. Mean results in the high spike were approximately five times the maximum reporting DL. Since the concentration of the high level round robin VHs spike was actually quite low, an additional single laboratory spike at a higher concentration of 7180 mg/kg was added. The results obtained for this additional spike can be found with the rest of the single laboratory data in Appendix B.

The Method Detection Limit for VH_{S6-10} , determined for the single laboratory data, was 9 mg/kg.

For the analysis of BTEX, six labs used GC with Mass Spectrometric detection (GC/MS), which is the preferred instrument. One lab used GC with Photo Ionization Detection (GC/PID). The GC/MS and the GC/PID results showed reasonable consistency for these samples. Two labs used GC with Flame Ionization Detection (GC/FID). GC/FID results were inconsistent with GC/MS or GC/PID results for these samples.

Method Detection Limits – Single Laboratory Results

Table 19: SINGLE LABORATORY METHOD DETECTION LIMIT RESULTS		
Method	Method Detection Limit (MDL)**	Units
EPH _{W10-19}	59	ug/L
EPH _{W19-32}	102	ug/L
EPH _{S10-19}	21	mg/kg
EPH _{S19-32}	9	mg/kg
VH _{W6-10}	13	ug/L
VH _{S6-10}	9	mg/kg

** MDL = Reliable Detection Limit (RDL) at 95% confidence, calculated as $(2 \times 1.90 \times \sigma)$ for 7 degrees of freedom.

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Method Detection Limits were experimentally determined as a component of the single laboratory data, and are summarized in Table 19. MDL's were calculated using the procedure recommended in the BC MELP Methods Manual, which is also equivalent to the procedure recommended by CAEAL. Eight matrix spikes were performed for each method. Spike concentrations were approximately three times the estimated MDL for each parameter. MDL's were calculated as indicated in the table.

Table 19 MDL's are provided only as a general indication of the sensitivity of the methods. MDL's can differ significantly among laboratories, and each laboratory must experimentally determine the appropriate MDL's that apply under their individual circumstances and conditions.

Acknowledgments

BCMELP would like to acknowledge all laboratories that participated in the 1998 BC Petroleum Hydrocarbon Methods Round Robin Study.

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APPENDIX A

COMPLETE ROUND ROBIN RESULTS

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Part 1.1: LEPHw-HEPHw Instrument Performance Check										Summary		
Relative Response	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	%RSD
Decane (nC10)	1.00	(4.06)	{0.82}	0.98	1.14	{1.00}	0.89	0.99	0.96	6	0.99	8.3%
Naphthalene	1.05	1.07	{0.88}	1.01	(2.17)	{1.11}	0.89	1.08	1.06	6	1.03	7.0%
Dodecane (nC12)	1.00	*	{0.87}	0.97	(co-elute)	{0.99}	0.94	*	0.99	4	0.97	2.8%
Hexadecane (nC16)	0.99	1.00	{0.98}	0.99	1.00	{1.04}	0.94	0.99	1.04	7	0.99	2.8%
Phenanthrene	1.03	1.05	{1.03}	1.07	0.94	{1.14}	1.01	1.09	1.12	7	1.04	5.7%
Nonadecane (nC19)	0.99	1.01	{1.00}	0.99	1.00	{1.00}	1.00	0.99	1.01	7	1.00	0.8%
Eicosane (nC20)	1.00	1.00	{1.00}	1.00	1.00	{1.00}	1.00	1.00	1.00	7	1.00	n/a
Pyrene	1.06	1.07	{1.00}	1.09	1.07	{1.03}	1.06	1.10	1.09	7	1.08	1.5%
Benzo(a)pyrene	0.92	0.80	{0.22}	*	0.59	{0.10}	0.69	1.04	0.88	6	0.82	19.6%
Triacontane (nC30)	0.98	*	{0.24}	1.07	0.98	{0.09}	0.69	1.01	0.81	6	0.92	15.5%
Dotriacontane (nC32)	0.96	0.87	{0.14}	1.06	0.78	{0.09}	0.68	0.99	0.77	7	0.87	16.0%

Sample Description: Relative GC-FID response for each compound (at 100ug/ml in solution) against nC20 is reported.

Key to excluded results:

{##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method.

(##) Data excluded based on technical information supplied by the laboratory:

-lab 2, decane :excluded due to co-elution with solvent peak.

-lab 6.; naphthalene and nC12 excluded due to co-elution with each other.

* Data not supplied by participating laboratory.

n/a Not applicable.

Part 1.2: LEPHw-HEPHw Method Performance Check										Summary		
Spike Recovery	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	%RSD
Decane (nC10)	66.3%	(78%)	{63.4%}	61.1%	60.2%	{56.8%}	54.0%	50.4%	37.5%	6	54.9%	18.6%
Naphthalene	84.7%	124%	{63.3%}	99.7%	(69.5%)	{84.2%}	65.8%	71.4%	51.4%	6	82.7%	31.3%
Dodecane (nC12)	79.4%	*	{64.7%}	74.6%	(co-elute)	{100%}	68.0%	*	55.0%	4	69.3%	15.3%
Hexadecane (nC16)	95.7%	123%	{70.4%}	103%	83.4%	{70.6%}	76.5%	86.6%	77.0%	7	92.1%	18.1%
Phenanthrene	96.7%	123%	{70.4%}	103%	84.0%	{39.7%}	78.0%	87.4%	79.7%	7	93.1%	17.1%
Nonadecane (nC19)	96.2%	124%	{73.2%}	107%	95.3%	{23.3%}	71.8%	87.6%	81.9%	7	94.7%	17.9%
Eicosane (nC20)	96.3%	123%	{72.8%}	108%	94.5%	{15.7%}	74.7%	87.6%	83.2%	7	95.4%	17.0%
Pyrene	96.3%	121%	{72.2%}	98.8%	93.3%	{10.9%}	76.5%	87.6%	84.1%	7	93.9%	15.0%
Benzo(a)pyrene	96.3%	124%	{72.6%}	*	93.4%	{36.4%}	122%	90.8%	72.5%	6	99.9%	19.9%
Triacontane (nC30)	96.3%	*	{71.5%}	97.8%	90.3%	{70.2%}	127%	87.5%	70.5%	6	94.8%	19.3%
Dotriacontane (nC32)	92.4%	125%	{120%}	95.6%	91.3%	{70.2%}	124%	87.3%	69.5%	7	97.8%	20.4%

Sample Description: Percent recovery for each compound is reported for a 200 ug/L aqueous spike sample.

Key to excluded results:

{##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method.

(##) Data excluded based on technical information supplied by the laboratory, or due to suspected calculation error:

-lab 2, decane :excluded due to co-elution with solvent peak.

-lab 6.; naphthalene and nC12 excluded due to co-elution with each other.

* Data not supplied by participating laboratory.

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Part 1.3: LEPHw-HEPHw Low Level Method Spike										Summary		
EPH Data (ug/L)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	%RSD
EPHw(10-19)	2737	4786	{2713}	2977	3934	{3427}	2710	2938	1950	7	3147	29.5%
EPHw(19-32)	645	695	{437}	595	630	{36}	557	539	393	7	579	16.9%
LEPHw	2733	4783	n/a	2974	3930	{3420}	2706	2930	1948	7	3143	29.5%
HEPHw	644	695	n/a	595	630	{36}	557	538	393	7	579	16.9%
PAH Data (ug/L)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	% RSD
Naphthalene	1.11	1.20	*	1.53	2.00	2.8	1.68	2.60	0.86	8	1.72	40.7%
Acenaphthene	<0.05	0.06	*	<0.49	<0.50	0.5	0.46	0.98	0.44	5	0.49	67.1%
Phenanthrene	1.55	0.33	*	0.90	1.30	1.7	1.40	2.26	0.94	8	1.30	45.0%
Fluorene	0.73	0.33	*	0.56	0.80	1.2	0.64	1.58	0.40	8	0.78	53.8%
Anthracene	0.19	0.03	*	<0.1	<0.50	<0.2	<0.3	<	<0.4	2	0.11	103%
Acridine	<0.5	0.52	*	<0.81	<0.50	0.7	<0.3	<	<0.4	5	<	n/a
Fluoranthene	<0.2	0.01	*	<0.01	<0.50	<0.2	<0.2	<	<0.4	6	<	n/a
Pyrene	0.17	0.06	*	0.08	<0.50	0.20	0.34	0.36	<0.4	6	0.20	62.8%
Benz(a)anthracene	<0.05	<0.01	*	0.03	<0.50	<0.2	<0.2	<	<0.4	6	<	n/a
Benzo(a)pyrene	<0.01	<0.01	*	<0.01	<0.50	<0.2	<0.3	<	<0.4	7	<	n/a
PAH Detector	MS	MS	n/a	MS	MS	MS	MS	MS	MS		MS	

Sample Description: EPH and PAH results are reported for an aqueous sample spiked with 5,000 ug/L of diesel.

Key to excluded results:

- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method.
- * Data not supplied by participating laboratory.
- n/a Not applicable

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Part 1.4: LEPHw-HEPHw High Level Method Spike										Summary		
EPH Data (ug/L)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	%RSD
EPHw(10-19)	18433	12451	{14579}	16710	20885	{16437}	15710	17301	12786	7	16325	18.4%
EPHw(19-32)	3608	3376	{1125}	2974	3377	{1999}	2163	2758	2592	7	2978	17.2%
LEPHw	18412	12443	n/a	16693	20863	{16409}	15684	17262	12770	7	16304	18.4%
HEPHw	3607	3376	n/a	2973	3376	{1999}	2162	2756	2592	7	2978	17.2%
PAH Data (ug/L)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	% RSD
Naphthalene	8.80	5.30	*	7.84	8.90	10	12.40	16.86	6.34	8	9.56	38.4%
Acenaphthene	<3	0.32	*	<2.71	2.30	2.2	2.95	5.12	2.82	6	2.62	59.1%
Fluorene	4.03	0.66	*	3.39	4.90	5.1	3.25	6.30	2.24	8	3.73	47.4%
Phenanthrene	7.82	1.70	*	5.18	5.90	7.1	7.29	11.20	4.66	8	6.36	43.4%
Anthracene	0.90	0.15	*	<0.56	0.70	<0.2	<0.3	<	<0.4	4	<	n/a
Acridine	<0.5	0.29	*	<5.59	<0.50	3.7	<0.3	<	<0.4	6	<	n/a
Fluoranthene	<0.2	0.05	*	<0.07	<0.50	<0.2	<0.2	<	<0.4	7	<	n/a
Pyrene	0.76	0.21	*	0.61	0.79	<0.2	1.17	1.56	<0.4	6	0.85	54.8%
Benz(a)anthracene	<0.05	0.01	*	<0.01	<0.50	<0.2	<0.2	<	<0.4	7	<	n/a
Benzo(a)pyrene	<0.01	0.01	*	<0.01	<0.50	<0.2	<0.3	<	<0.4	7	<	n/a
PAH Detector	MS	MS	n/a	MS	MS	MS	MS	MS	MS		MS	

Sample Description: EPH and PAH results are reported for an aqueous sample spiked with 25,000 ug/L of diesel.

Key to excluded results:

- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method.
- * Data not supplied by participating laboratory.
- n/a Not applicable

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Part 2.1: LEPHs-HEPHs Instrument Performance Check										Summary		
Relative Response	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	%RSD
Decane (nC10)	1.01	(3.52)	[0.82]	0.98	1.08	{1.00}	0.89	0.99	0.96	6	0.98	6.3%
Naphthalene	1.06	1.07	[0.88]	1.01	(2.12)	{1.10}	0.89	1.08	1.06	6	1.03	6.9%
Dodecane (nC12)	1.01	*	[0.87]	*	(co-elute)	{1.10}	0.94	0.98	0.99	4	0.98	3.1%
Hexadecane (nC16)	0.99	1.00	[0.98]	0.99	0.99	{0.99}	0.94	0.99	1.04	7	0.99	2.8%
Phenanthrene	1.03	1.05	[1.03]	1.07	0.98	{1.04}	1.01	1.09	1.12	7	1.05	4.6%
Nonadecane (nC19)	0.99	1.00	[1.00]	0.99	1.01	{1.14}	1.00	0.99	1.01	7	1.00	0.8%
Eicosane (nC20)	1.00	1.00	[1.00]	1.00	1.00	{1.00}	1.00	1.00	1.00	7	1.00	n/a
Pyrene	1.06	1.08	[1.00]	1.09	1.08	{1.03}	1.00	1.10	1.09	7	1.07	3.3%
Benzo(a)pyrene	0.92	0.79	[0.22]	*	0.86	{0.10}	0.69	1.04	0.88	6	0.87	13.6%
Triacontane (nC30)	1.00	*	[0.24]	*	1.04	{0.09}	0.68	0.99	0.81	5	0.90	17.2%
Dotriacontane (nC32)	1.05	0.86	[0.14]	1.06	0.89	{0.06}	0.68	0.99	0.77	7	0.90	16.1%
Extraction Method	Soxhlet	Soxhlet	Sonic.	Soxhlet	Soxhlet	Soxhlet	Soxhlet	Soxhlet	Soxhlet		Soxhlet	
Solvent	1:1H/A	1:1H/A	1:3H/A	1:1H/A	1:1H/A	1:1H/A	1:1H/A	DCM	1:1H/A		1:1H/A	
Hours of extraction	24	16	1	16	16	16	16	16	16			

Sample Description: Relative GC-FID response for each compound (at 100ug/ml in solution) against nC20 is reported.

Key to excluded results:

- [##] Data excluded due to failure to use extraction solvent or extraction mechanism specified in BCMELP method (should be hexane:acetone Soxhlet).
- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method
- (##) Data excluded based on technical information supplied by the laboratory:
 - lab 2, decane :excluded due to co-elution with solvent peak.
 - lab 6,; naphthalene and nC12 excluded due to co-elution with each other.
- * Data not supplied by participating laboratory.
- n/a Not applicable.

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Part 2.2: LEPHs-HEPHs Method Performance Check										Summary		
Spike Recovery	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	%RSD
Decane (nC10)	7.2%	30.0%	[80.1%]	*	94.2%	{0.13%}	*	[92.7%]	35.2%	4	41.6%	89.0%
Naphthalene	19.4%	56.5%	[63.2%]	30.1%	(74.3%)	{3.8%}	*	[98.4%]	68.5%	4	43.6%	52.2%
Dodecane (nC12)	27.0%	*	[83.4%]	*	(co-elute)	{9.4%}	26.2%	[98.2%]	69.6%	3	40.9%	60.7%
Hexadecane (nC16)	86.0%	88.0%	[87.6%]	92.4%	81.6%	{9.4%}	57.5%	[101%]	80.0%	6	80.9%	15.2%
Phenanthrene	87.5%	91.1%	[70.3%]	86.9%	63.0%	{77.4%}	41.3%	[99.6%]	81.0%	6	75.1%	25.8%
Nonadecane (nC19)	87.0%	96.1%	[87.3%]	95.8%	91.2%	{33.3%}	51.5%	[101%]	80.2%	6	83.6%	20.1%
Eicosane (nC20)	93.2%	97.6%	[86.9%]	97.3%	86.1%	{14.6%}	45.4%	[101%]	80.5%	6	83.4%	23.7%
Pyrene	89.7%	95.7%	[65.6%]	76.0%	43.3%	{6.3%}	23.5%	[99.6%]	80.9%	6	68.2%	41.8%
Benzo(a)pyrene	*	102.0%	[60.2%]	*	42.1%	{3.9%}	*	[102%]	60.6%	3	68.2%	45.0%
Triacontane (nC30)	92.9%	*	[34.0%]	*	91.6%	{7.8%}	*	[104%]	79.6%	3	88.0%	8.4%
Dotriacontane (nC32)	88.3%	108.0%	[64.0%]	87.0%	116%	{1.6%}	*	[104%]	74.2%	5	94.7%	17.9%
Extraction Method	Soxhlet	Soxhlet	Sonic.	Soxhlet	Soxhlet	Soxhlet	Soxhlet	Soxhlet	Soxhlet		Soxhlet	
Solvent	1:1H/A	1:1H/A	1:3H/A	1:1H/A	1:1H/A	1:1H/A	1:1H/A	DCM	1:1H/A		1:1H/A	
Hours of extraction	24	16	1	16	16	16	16	16	16			

Sample Description: Percent recovery for each compound is reported for a 10-50 mg/kg clean sand spike sample.

Note: Low Round Robin recoveries for this sample were due to spiking problems that were addressed prior to single-lab data generation. Version 2.1 of the BCMELP methods corrects this problem.

Key to excluded results:

- [##] Data excluded due to failure to use extraction solvent or extraction mechanism specified in BCMELP method (should be hexane:acetone Soxhlet).
- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method
- (##) Data excluded based on technical information supplied by the laboratory:
 - lab 2 data was included into statistical analyses after receiving the draft round robin data.
 - lab 6.; naphthalene and nC12 excluded due to co-elution with each other.
- * Data not supplied by participating laboratory.
- n/a Not applicable.

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Part 2.3: LEPHs-HEPHs Analysis of Reference Material 1 (RTC TPH 355-100)										Summary		
EPH Data (mg/kg)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	%RSD
EPHs(10-19)	3481	3428	[2252]	2713	3588	{8341}	3510	[3471]	3150	6	3312	9.9%
EPHs(19-32)	5317	5463	[963]	4094	5087	{1018}	6317	[4752]	3953	6	5038	17.7%
LEPHs	3469	3416	n/a	2708	3580	{8333}	3497	[3461]	3144	6	3302	9.9%
HEPHs	5316	5462	n/a	4093	5086	{1017}	6316	[4752]	3953	6	5038	17.7%
PAH Data (mg/kg)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	% RSD
Naphthalene	4.87	4.70	*	2.35	3.70	3	5.74	4.94	3.16	8	4.06	28.9%
Phenanthrene	7.44	7.40	*	3.39	4.45	5	7.41	4.83	2.82	8	5.34	34.8%
Pyrene	0.78	0.93	*	0.53	0.63	0.8	1.31	0.27	0.27	8	0.69	50.3%
Benz(a)anthracene	0.08	0.18	*	0.04	<0.20	0.13	<0.1	<	<0.1	4	0.11	55.5%
Benzo(b)fluoranthene	0.05	0.04	*	0.03	<0.20	<0.05	<0.05	<	<0.1	3	0.04	20.8%
Benzo(k)fluoranthene	0.01	0.04	*	0.01	<0.20	<0.05	<0.05	<	<0.1	3	0.02	75.8%
Benzo(a)pyrene	0.05	0.06	*	0.03	<0.20	<0.05	<0.05	<	<0.1	3	0.05	32.8%
Indeno(1,2,3-cd)pyrene	0.02	0.02	*	0.02	<0.20	<0.05	<0.05	<	<0.1	3	0.02	2.8%
Dibenz(a,h)anthracene	0.01	0.01	*	0.01	<0.20	<0.05	<0.05	<	<0.1	3	0.01	10.8%
Extraction Method	Soxhlet	Soxhlet	Sonic.	Soxhlet	Soxhlet	Soxhlet	Soxhlet	Soxhlet	Soxhlet		Soxhlet	
Solvent	1:1H/A	1:1H/A	1:3H/A	1:1H/A	1:1H/A	1:1H/A	1:1H/A	DCM	1:1H/A		1:1H/A	
Hours of extraction	24	16	1	16	16	16	16	16	16			
PAH Detector	MS	MS	n/a	MS	MS	MS	MS	MS	MS		MS	

Sample Description: EPH and PAH results are reported for the RTC TPH 355-100 reference material.

Key to excluded results:

- [##] Data excluded due to failure to use extraction solvent or extraction mechanism specified in BCMELP method (should be hexane:acetone Soxhlet).
- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method
- (##) Data excluded based on technical information supplied by the laboratory:
-lab 2 data was included into statistical analyses after receiving the draft round robin data.
- * Data not supplied by participating laboratory.
- n/a Not applicable.

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Part 2.4: LEPHs-HEPHs Analysis of Reference Material 2 (NRC Canada HS3B)										Summary		
EPH Data (mg/kg)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	% RSD
EPHs(10-19)	473	422	[179.8]	286	(17)	{703}	374	[370]	373	5	385	18.0%
EPHs(19-32)	2502	2748	[369.2]	2089	(278)	{359}	3979	[3321]	2405	5	2745	26.6%
LEPHs	451	402	n/a	275	(5)	{683}	353	[354]	362	5	369	17.6%
HEPHs	2454	2706	n/a	2065	(246)	{351}	3928	[3294]	2383	5	2707	26.6%
PAH Data (mg/kg)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	Lab10	(n)	Mean	% RSD
Naphthalene	2.01	1.8	*	0.78	1.14	1.6	2.43	1.54	1.67	8	1.62	31.1%
Phenanthrene	19.6	18	*	9.68	11.2	18	18.0	14.88	9.91	8	14.9	27.4%
Pyrene	16.7	16	*	9.00	11.2	15	19.5	14.32	8.06	8	13.7	28.9%
Benz(a)anthracene	7.29	8.5	*	3.20	4.89	7	7.79	5.02	3.11	8	5.85	35.6%
Benzo(b)fluoranthene	9.42	5.45	*	4.25	6.61	6	11.8	5.25	3.19	8	6.50	43.5%
Benzo(k)fluoranthene	3.72	5.45	*	1.82	2.17	3	4.87	2.05	2.92	8	3.25	41.1%
Benzo(a)pyrene	5.52	4.3	*	2.72	3.65	4	4.87	2.73	2.53	8	3.79	28.8%
Indeno(1,2,3-cd)pyrene	4.31	2.9	*	2.24	2.91	2.2	1.20	<	1.81	7	2.51	39.6%
Dibenz(a,h)anthracene	1.09	0.22	*	0.59	0.67	0.6	0.29	<	0.51	7	0.57	50.2%
Extraction Method	Soxhlet	Soxhlet	Sonic.	Soxhlet	Soxhlet	Soxhlet	Soxhlet	Soxhlet	Soxhlet		Soxhlet	
Solvent	1:1H/A	1:1H/A	1:3H/A	1:1H/A	1:1H/A	1:1H/A	1:1H/A	DCM	1:1H/A		1:1H/A	
Hours of extraction	24	16	1	16	16	16	16	16	16			
PAH Detector	MS	MS		MS	MS	MS	MS	MS	MS		MS	

Sample Description: EPH and PAH results are reported for NRC Canada HS3B certified reference material.

Key to excluded results:

- [##] Data excluded due to failure to use extraction solvent or extraction mechanism specified in BCMELP method (should be hexane:acetone Soxhlet).
- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method
- (##) Data excluded based on technical information supplied by the laboratory:
 - lab 2 data includes corrections made following distribution of draft round robin data to participants.
 - lab 6, EPHs(10-19) and EPHs(19-32): calculation or calibration error suspected.
- * Data not supplied by participating laboratory.
- n/a Not applicable.

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Part 3.1: VPHw Instrument and Method Performance Check									Summary		
Relative Response	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	(n)	Mean	% RSD
Hexane (nC6) ¹	0.96	0.87	1.06	0.62	1.29	{0.67}	[2.61]	{1.52}	5	0.96	25.8%
Benzene ¹	1.08	0.98	1.13	1.02	1.24	{0.59}	[0.72]	{2.30}	5	1.09	9.4%
Toluene ¹	1.04	0.99	1.01	1.01	1.12	{0.92}	[0.86]	{1.54}	5	1.04	4.9%
Octane (nC8) ¹	0.98	0.98	0.90	0.52	1.12	{0.43}	[2.75]	*	5	0.90	25.1%
Ethylbenzene ¹	1.01	0.99	0.93	0.99	1.04	{0.89}	[1.02]	{0.69}	5	0.99	4.1%
m,p-Xylene ¹	1.00	1.00	1.00	1.00	1.00	{1.00}	[1.00]	{1.00}	5	1.00	n/a
o-Xylene ²	1.10	0.99	(0.59)	1.09	1.16	{3.51}	[0.95]	{0.71}	4	1.08	6.4%
1,2,4-Trimethylbenzene ²	1.00	1.00	1.00	1.00	1.00	{1.00}	[1.00]	{1.00}	5	1.00	n/a
Decane (nC10) ²	0.89	0.72	(co-elute)	0.51	0.90	{3.20}	[2.42]	{1.22}	4	0.76	24.0%
Sample Introduction	P&T	P&T	P&T	P&T	P&T	P&T	HS	P&T		P&T	

Sample Description: Relative GC-FID response for each compound (at 100 ug/L) against m,p-Xylene or 1,2,4-Trimethylbenzene is reported.

Key to excluded results:

- [##] Data excluded due to failure to use sample introduction mechanism specified in BCMELP method (should be Purge and Trap).
- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method
- (##) Data excluded based on technical information supplied by the laboratory:
-lab 4, o-xylene and decane: excluded due to co-elution with each other.
- ¹ Relative response calculated against m,p-Xylene.
- ² Relative response calculated against 1,2,4-Trimethylbenzene.
- * Data not supplied by participating laboratory.
- n/a Not applicable.

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Part 3.2: VPHw Low Level Method Spike									Summary		
VH Data (ug/L)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	(n)	Mean	% RSD
VHw(6-oXylene)	878	893	670	728	1293	{586}	[1856]	(15969)	5	892	27.3%
VHw(oXylene-10)	176	145	42	119	103	{33.1}	[259]	(1972)	5	117	42.9%
VHw(6-10)	1054	1038	712	847	1396	{619}	[2115]	(17940)	5	1010	25.6%
VPHw	827	884	(377)	621	1190	{340}	[1724]	(17673)	4	880	26.7%
BTEX Data (ug/L)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	(n)	Mean	% RSD
Benzene	15.3	17.0	(115)	16.5	17.2	15.0	(54)	21.4	6	17.1	13.5%
Toluene	108.0	62.0	(110)	94.8	75.0	99.0	(184)	101.1	6	90.0	19.6%
Ethylbenzene	20.3	15.0	(20)	22.3	23.8	20.0	(33)	26.8	6	21.4	18.7%
m,p-Xylene	60.0	41.0	(60)	66.9	62.5	120.0	(78)	87.2	6	72.9	37.6%
o-Xylene	23.7	19.0	(30)	25.9	27.8	25.0	(42)	30.8	6	25.4	15.7%
Sample Introduction	P&T	P&T	P&T	P&T	P&T	P&T	HS	P&T		P&T	
BTEX Detector	MS	PID	FID	MS	MS	MS	FID	MS		MS	

Sample Description: VH and BTEX results are reported for an aqueous sample spiked with 2,000 ug/L of gasoline.

Key to excluded results:

- [##] Data excluded due to failure to use sample introduction mechanism specified in BCMELP method (should be Purge and Trap).
- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method
- (##) Data excluded based on technical information supplied by the laboratory, or due to suspected calculation error:
 - lab 4 & lab 8, BTEX and VPH results: excluded due to use of Flame Ionization Detector.
 - lab 6, BTEX and VPH results: includes corrections made following distribution of draft round robin data to participants.
 - lab 9, VH and VPH results: calculation or calibration error suspected, instrument relative response requirements not met.

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Part 3.3: VPHw High Level Method Spike									Summary		
VH Data (ug/L)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	(n)	Mean	% RSD
VHw(6-oXylene)	4291	4032	4451	3601	7064	{1641}	[8917]	(141934)	5	4688	29.2%
VHw(oXylene-10)	1159	757	350	664	694	{173}	[1249]	(7456)	5	725	39.9%
VHw(6-10)	5450	4789	4800	4265	7758	{1815}	[10166]	(149390)	5	5412	25.4%
VPHw	4318	4000	(4465)	3132	n/a	n/a	[8243]	(148053)	3	3817	16.1%
BTEX Data (ug/L)	Lab1	Lab2	Lab4	Lab5	Lab6	Lab7	Lab8	Lab9	(n)	Mean	% RSD
Benzene	77	72	(115)	83	(over)	(10.2)	(254)	107	4	84.6	18.4%
Toluene	535	350	(110)	474	(over)	(over)	(868)	506	4	466	17.5%
Ethylbenzene	102	65	(20)	112	(over)	(over)	(175)	134	4	103	27.9%
m,p-Xylene	300	220	(60)	335	(over)	(over)	(410)	436	4	323	27.7%
o-Xylene	118	82	(30)	129	(over)	(over)	(216)	154	4	121	24.8%
Sample Introduction	P&T	P&T	P&T	P&T	P&T	P&T	HS	P&T		P&T	
BTEX Detector	MS	PID	FID	MS	MS	MS	FID	MS		MS or PID	

Sample Description: VH and BTEX results are reported for an aqueous sample spiked with 10,000 ug/L of gasoline.

Key to excluded results:

- [##] Data excluded due to failure to use sample introduction mechanism specified in BCMELP method (should be Purge and Trap).
- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method.
- (##) Data excluded based on technical information supplied by the laboratory: or due to suspected calculation error:
 - lab 4 & lab 8, BTEX and VPH results: excluded due to use of Flame Ionization Detector.
 - lab 6, BTEX results: reported as out of calibration range.
 - lab 7, BTEX results: 5 results reported as out of calibration range: calculation or calibration error suspected for benzene.
 - lab 9, VH and VPH results: calculation or calibration error suspected, instrument relative response requirements not met.

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Part 4.1: VPHs Instrument Performance Check										Summary DI			Summary P&T		
Relative Response	Lab1	Lab4	Lab5	Lab8	Lab9	Lab10	Lab2	Lab6	Lab7	(n)	Mean	%RSD	(n)	Mean	% RSD
Hexane (nC6) ¹	0.75	{0.79}	0.87	*	0.80	0.70	0.88	1.32	{0.75}	4	0.78	9.0%	2	1.10	28.1%
Benzene ¹	1.00	{0.85}	1.07	1.00	0.96	0.95	1.05	1.23	{0.37}	5	1.00	4.9%	2	1.14	11.1%
Toluene ¹	1.02	{0.81}	1.04	0.98	0.95	0.99	1.02	1.12	{1.00}	5	0.99	3.5%	2	1.07	6.5%
Octane (nC8) ¹	0.92	{0.88}	0.93	0.94	0.89	0.88	0.90	1.05	{0.57}	5	0.91	2.7%	2	0.97	11.2%
Ethylbenzene ¹	0.99	{1.03}	1.01	0.99	0.93	1.01	1.01	1.04	{0.94}	5	0.99	3.1%	2	1.02	2.4%
m,p-Xylene ¹	1.00	{1.00}	1.00	1.00	1.00	1.00	1.00	1.00	{1.00}	5	1.00	n/a	2	1.00	n/a
o-Xylene ²	1.08	{2.43}	1.10	1.10	1.05	1.04	1.07	1.16	{1.61}	5	1.07	2.6%	2	1.12	5.5%
1,2,4-Trimethylbenzene ²	1.00	{1.00}	1.00	1.00	1.00	1.00	1.00	1.00	{1.00}	5	1.00	n/a	2	1.00	n/a
Decane (nC10) ²	0.91	{2.17}	0.85	0.94	0.87	0.86	0.73	0.84	{1.15}	5	0.89	4.3%	2	0.78	10.0%
Sample Introduction	DI	DI	DI	DI	DI	DI	P&T	P&T	P&T		DI			P&T	

Sample Description: Relative GC-FID response for each compound (at 50 ug/L in methanol) against m,p-Xylene or 1,2,4-Trimethylbenzene is reported.

Note: **Direct Injection (DI) is the reference technique for the BCMELP VH in sediment method.** VH results by Purge and Trap (P&T) are summarized separately. P&T can be an allowable alternative to Direct Injection, where method equivalence is shown, and where required method QC steps are conformed with.

Key to excluded results:

{##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method

¹ Relative response calculated against m,p-Xylene.

² Relative response calculated against 12,4-Trimethylbenzene.

* Data not supplied by participating laboratory.

n/a Not applicable.

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Part 4.2: VPHs Method Performance Check										Summary DI			Summary P&T		
Spike Recovery	Lab1	Lab4	Lab5	Lab8	Lab9	Lab10	Lab2	Lab6	Lab7	(n)	Mean	%RSD	(n)	Mean	%RSD
Hexane (nC6)	98.5%	{95.6%}	98.4%	*	109%	104%	91.3%	103%	{2.49%}	4	102%	4.7%	2	97%	8.6%
Benzene	98.7%	{84.0%}	102%	109%	108%	105%	100%	108%	{67.1%}	5	104%	4.1%	2	104%	5.3%
Toluene	98.2%	{87.9%}	101%	113%	110%	104%	102%	102%	{80.0%}	5	105%	5.9%	2	102%	0.1%
Octane (nC8)	97.0%	{62.8%}	101%	109%	108%	103%	101%	105%	{52.4%}	5	103%	4.8%	2	103%	3.1%
Ethylbenzene	101%	{55.9%}	103%	111%	108%	105%	102%	97.1%	{85.0%}	5	106%	3.9%	2	100%	3.5%
m,p-Xylene	99.9%	{48.2%}	103%	111%	108%	106%	103%	103%	{85.1%}	5	106%	4.0%	2	103%	0.2%
o-Xylene	99.5%	{46.2%}	104%	111%	108%	107%	102%	101%	{85.7%}	5	106%	4.2%	2	102%	0.9%
1,2,4-Trimethylbenz.	104%	{67.7%}	108%	115%	107%	110%	103%	108%	{84.4%}	5	109%	3.5%	2	105%	3.6%
Decane (nC10)	97.5%	*	106%	111%	107%	104%	106%	114%	{86.1%}	5	105%	4.9%	2	110%	4.8%
Sample Extraction	MeOH	MeOH	MeO H DI	MeOH	MeOH	MeOH	MeOH	MeOH	MeOH		MeOH			MeOH	
Sample Introduction	DI	DI	DI	DI	DI	DI	P&T	P&T	P&T		DI			P&T	

Sample Description: Percent recovery for each compound is reported for a 110 mg/kg clean sand spike sample.

Note: **Direct Injection (DI) is the reference technique for the BCMELP VH in sediment method.** VH results by Purge and Trap (P&T) are summarized separately. P&T can be an allowable alternative to Direct Injection, where method equivalence is shown, and where required method QC steps are conformed with.

Key to excluded results:

{##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method

* Data not supplied by participating laboratory.

n/a Not applicable.

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Part 4.3: VPHs Low Level Method Spike										Summary DI		Summary P&T			
VH Data (mg/kg)	Lab1	Lab4	Lab5	Lab8	Lab9	Lab10	Lab2	Lab6	Lab7	(n)	Mean	%RSD	(n)	Mean	%RSD
VHs(6-oXylene)	82.0	{52.5}	128	71.0	103	95.1	101	193	{49.2}	5	95.7	22.6%	2	147	44.2%
VHs(oXylene-10)	13.8	{0}	19.1	13.0	15.9	15.8	17.3	13.4	{10.4}	5	15.5	15.3%	2	15.3	17.9%
VHs(6-10)	95.8	{52.5}	147	84.0	119	111	119	207	{59.5}	5	111	21.5%	2	163	38.3%
VPHs	62.7	(38.5)	117	(84.0)	81.3	87.8	101	174	{38.3}	4	87.3	26.0%	2	138	37.4%
BTEX Data (mg/kg)	Lab1	Lab4	Lab5	Lab8	Lab9	Lab10	Lab2	Lab6	Lab7	(n)	Mean	% RSD	(n)	Mean	% RSD
Benzene	2.41	(14)	2.12	(0.01)	3.23	1.69	2	2.31	1.5	4	2.36	27.5%	3	1.94	21.1%
Toluene	14.8	(<)	12.0	(0.01)	14.7	9.38	6.7	13.7	6.9	4	12.7	20.2%	3	9.10	43.8%
Ethylbenzene	3.05	(<)	3.09	(<0.01)	3.57	2.24	1.8	3.19	1.8	4	2.99	18.5%	3	2.26	35.5%
m,p-Xylene	9.33	(<)	8.60	(<0.01)	11.7	7.28	4.7	9.78	11	4	9.22	19.9%	3	8.49	39.3%
o-Xylene	3.52	(<)	3.57	(<0.01)	4.28	2.45	2.2	3.63	co-elute	4	3.46	21.8%	2	2.92	34.7%
Styrene	<0.05	(<)	<	(<)	<	0.05	<0.02	<0.5	<20	6	<	n/a	3	<	n/a
Sample Extraction	MeOH	MeOH	MeOH	MeOH	MeOH	MeOH	MeOH	MeOH	MeOH		MeOH			MeOH	
Sample Introduction	DI	DI	DI	DI	DI	DI	P&T	P&T	P&T		DI			P&T	
BTEX Detector	MS	FID	MS	FID	MS	HS/MS	PID	MS	MS		MS			MS or PID	

Sample Description: VH and BTEX results are reported for a spike of 250 mg/kg gasoline into a clean sand sample.

Note: **Direct Injection (DI) is the reference technique for the BCMELP VH in sediment method.** VH results by Purge and Trap (P&T) are summarized separately. P&T can be an allowable alternative to Direct Injection, where method equivalence is shown, and where required method QC steps are conformed with.

Key to excluded results:

- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method.
- (##) Data excluded based on technical information supplied by the laboratory:
 - lab 4 & lab 8, BTEX and VPH results: excluded due to use of Flame Ionization Detector.
 - lab 7, ortho-Xylene results, excluded due to co-elution with m,p-Xylene.
- n/a Not applicable.

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Part 4.4: VPHs Medium Level Method Spike										Summary DI			Summary P&T		
VH Data (mg/kg)	Lab1	Lab4	Lab5	Lab8	Lab9	Lab10	Lab2	Lab6	Lab7	(n)	Mean	%RSD	(n)	Mean	%RSD
VHs(6-oXylene)	379	{288}	460	443	510	451	441	979	{293}	5	449	10.5%	2	710	53.6%
VHs(oXylene-10)	84.5	{6.4}	80.5	95.0	89.8	94.7	71.6	103	{48.7}	5	88.9	7.1%	2	87.3	25.4%
VHs(6-10)	463	{294}	541	538	600	546	513	1081	{342}	5	538	9.1%	2	797	50.4%
VPHs	337	{177}	429	(538)	507	415	443	954	{235}	4	422	16.5%	2	699	51.7%
BTEX Data (mg/kg)	Lab1	Lab4	Lab5	Lab8	Lab9	Lab10	Lab2	Lab6	Lab7	(n)	Mean	% RSD	(n)	Mean	% RSD
Benzene	10.2	(62)	9.02	(0.01)	5.74	11.0	7.1	14.1	5.2	4	8.99	25.7%	3	8.80	53.3%
Toluene	61.6	(45)	49.2	(0.06)	41.6	57.8	33.0	49.1	32	4	52.6	17.0%	3	38.0	25.2%
Ethylbenzene	12.5	(3)	12.3	(0.01)	9.05	13.2	6.7	17.8	8.7	4	11.8	15.8%	3	11.1	53.5%
m,p-Xylene	38.0	(6)	35.7	(0.04)	31.4	44.7	21.0	39.4	51	4	37.5	14.8%	3	37.1	40.7%
o-Xylene	14.6	(1)	14.3	(0.02)	10.8	15.0	8.4	20.3	co-elute	4	13.7	14.3%	2	14.4	58.6%
Styrene	<0.05	(<)	<	(<)	<	0.25	<0.02	<0.5	<20	6	<	n/a	3	<	n/a
Sample Extraction	MeOH	MeOH	MeOH	MeOH	MeOH	MeOH	MeOH	MeOH	MeOH		MeOH			MeOH	
Sample Introduction	DI	DI	DI	DI	DI	DI	P&T	P&T	P&T		DI			P&T	
BTEX Detector	MS	FID	MS	FID	MS	HS/MS	PID	MS	MS		All			All	

Sample Description: VH and BTEX results are reported for a spike of 1250 mg/kg gasoline into a clean sand sample.

Note: Direct Injection (DI) is the reference technique for the BCMELP VH in sediment method. VH results by Purge and Trap (P&T) are summarized separately. P&T can be an allowable alternative to Direct Injection, where method equivalence is shown, and where required method QC steps are conformed with.

Key to excluded results:

- {##} Data excluded due to failure to meet instrument relative response requirements specified in BCMELP method.
- (##) Data excluded based on technical information supplied by the laboratory:
 - lab 4 & lab 8, BTEX and VPH results: excluded due to use of Flame Ionization Detector.
 - lab 7, ortho-Xylene results, excluded due to co-elution with m,p-Xylene.
- n/a Not applicable.

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APPENDIX B

SINGLE LABORATORY RESULTS

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EPH in Waters – Instrument Performance Check Relative Response Data (versus nC20)										
units = relative area	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD
Decane	0.98	0.99	0.98	0.99	0.99	0.98	1.00	0.98	0.99	0.8%
Naphthalene	1.01	1.02	1.03	1.03	1.02	1.00	1.02	1.01	1.02	1.0%
Dodecane	0.99	0.99	0.99	1.01	1.02	1.01	1.03	1.020	1.01	1.6%
Hexadecane	0.98	0.99	0.98	0.99	1.00	0.99	1.00	1.000	0.99	0.8%
Phenanthrene	1.00	1.00	1.02	0.98	1.01	1.02	1.00	1.000	1.00	1.3%
Nonadecane	0.99	0.99	0.99	0.99	0.97	0.99	0.99	0.99	0.99	0.7%
Eicosane	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	n/a
Pyrene	1.04	1.05	1.02	0.94	1.03	1.03	0.98	1.01	1.01	3.6%
Triacontane	1.05	1.03	1.02	0.99	0.96	0.95	0.88	0.96	0.98	5.6%
Dotriacontane	1.03	1.01	0.88	0.95	0.88	0.87	0.80	0.93	0.92	8.4%

Corresponds with BCMELP Round Robin part 1.1.

EPH in Waters – Method Performance Check Recovery Data										
units = % recovery	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD
Decane	92%	83%	84%	87%	88%	79%	87%	85%	85.6%	4.5%
Naphthalene	108%	106%	100%	99%	104%	91%	103%	100%	101%	5.2%
Dodecane	106%	100%	98%	96%	102%	89%	100%	98%	98.6%	5.0%
Hexadecane	111%	112%	102%	100%	108%	95%	103%	105%	105%	5.5%
Phenanthrene	113%	112%	102%	100%	109%	94%	105%	106%	105%	6.1%
Nonadecane	112%	112%	102%	101%	109%	95%	106%	106%	105%	5.6%
Eicosane	112%	112%	103%	102%	108%	94%	106%	106%	105%	5.6%
Pyrene	112%	111%	116%	103%	108%	91%	105%	104%	106%	7.1%
benzo(a)pyrene	117%	119%	108%	107%	114%	98%	112%	109%	111%	6.0%
Triacontane	117%	117%	107%	105%	112%	97%	112%	109%	110%	6.1%
Dotriacontane	107%	109%	102%	98%	105%	91%	104%	102%	102%	5.5%
Tetracontane	100%	99%	90%	90%	95%	85%	95%	94%	93.5%	5.3%

Corresponds with BCMELP Round Robin part 1.2.

EPH in Waters – Method Detection Limit Data													
units = ug/L	#1	#2	#3	#4	#5	#6	#7	#8	Mean	Std-Dev	Target*	Mean Recovery	MDL**
EPHw(10-19)	146	151	129	159	130	159	156	175	151	15.5	235	64 %	59
EPHw(19-32)	302	343	284	329	268	340	308	326	313	26.9	294	106 %	102

* Target was determined by analysis of spike solution for EPH parameters.

** MDL = Reliable Detection Limit (RDL) at 95% confidence, calculated as (2 x 1.90 x SD) for 7 degrees of freedom.

Note: Data presented demonstrated achievable MDL's. Individual labs must determine their own MDL's. No correspondence with BCMELP Round Robin Study.

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EPH/LEPH/HEPH in Waters - Low Level Spike (5000 ug/L diesel)										
units = ug/L	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD
EPHw(10-19)	3740	3490	3190	3790	3100	3510	3120	3130	3384	8.4%
EPHw(19-32)	570	660	500	580	570	530	570	540	565	8.3%
Naphthalene	1.11	0.87	0.78	0.92	0.91	1.05	0.93	0.75	0.91	13.3%
Acenaphthylene*	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	n/a
Acenaphthene*	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	n/a
Fluorene	0.73	0.69	0.67	0.72	0.72	0.72	0.74	0.67	0.71	3.8%
Phenanthrene	1.55	1.50	1.50	1.58	1.51	1.53	1.57	1.44	1.52	3.0%
Anthracene	0.19	0.22	0.20	0.23	0.22	0.21	0.22	0.19	0.21	7.2%
Fluoranthene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Pyrene	0.17	0.15	0.16	0.17	0.16	0.15	0.15	0.14	0.16	6.8%
Benz(a)anthracene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Chrysene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Benzo(b)fluoranthene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Benzo(k)fluoranthene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Benzo(a)pyrene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Indeno(123cd)pyrene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Benzo(ghi)perylene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Dibenz(ah)anthracene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Acridine*	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	n/a
LEPH (calculated)	3736	3487	3187	3787	3097	3506	3117	3127	3380	8.4%
HEPH (calculated)	570	660	500	580	570	530	570	540	565	8.3%

Corresponds with BCMELP Round Robin part 1.3.

* Values could not be determined due to unresolved interference.

EPH/LEPH/HEPH in Waters – High Level Spike (25000 ug/L diesel)										
units = ug/L	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD
EPHw(10-19)	18150	17590	18160	16410	16720	16360	17600	18430	17428	4.7%
EPHw(19-32)	3400	3360	3430	3090	3280	3170	3390	3610	3341	4.8%
Naphthalene	5.31	6.53	8.09	7.76	8.55	8.80	9.62	10.30	8.12	19.9%
Acenaphthylene*	<1	<1	<1	<1	<1	<1	<1	<1	<1	n/a
Acenaphthene*	<3	<3	<3	<3	<3	<3	<3	<3	<3	n/a
Fluorene	3.94	4.04	4.05	3.99	3.97	4.03	4.01	3.92	3.99	1.2%
Phenanthrene	7.82	7.94	7.78	7.73	7.67	7.82	7.68	7.46	7.74	1.8%
Anthracene	0.99	1.00	1.01	0.99	0.98	0.90	0.98	0.97	0.98	3.4%
Fluoranthene	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	n/a
Pyrene	0.72	0.76	0.76	0.78	0.75	0.76	0.77	0.75	0.76	2.3%
Benz(a)anthracene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Chrysene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Benzo(b)fluoranthene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Benzo(k)fluoranthene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Benzo(a)pyrene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Indeno(123cd)pyrene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Benzo(ghi)perylene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Dibenz(ah)anthracene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a
Acridine*	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	n/a
LEPH (calculated)	18132	17570	18139	16390	16699	16338	17578	18407	17407	4.8%
HEPH (calculated)	3399	3359	3429	3089	3279	3169	3389	3609	3340	4.8%

Corresponds with BCMELP Round Robin part 1.4.

* Values could not be determined due to unresolved interference.

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EPH in Solids – Instrument Performance Check Relative Response Data (versus nC20)										
units = relative area	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD
Decane	1.03	1.04	0.99	1.00	0.99	1.02	1.02	1.02	1.01	1.8%
Naphthalene	1.07	1.07	1.05	1.06	1.08	1.09	1.08	1.09	1.07	1.3%
Dodecane	0.98	1.01	0.99	0.99	1.01	1.02	1.01	1.01	1.00	1.4%
Hexadecane	1.00	1.02	0.97	1.00	1.01	1.00	1.01	1.01	1.00	1.5%
Phenanthrene	1.06	1.08	1.05	1.07	1.06	1.06	1.06	1.06	1.06	0.8%
Nonadecane	0.99	1.00	0.99	0.99	0.99	0.99	0.99	0.99	0.99	0.4%
Eicosane	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	n/a
Pyrene	1.06	1.10	1.08	1.08	1.06	1.09	1.09	1.08	1.08	1.3%
Benzo(a)pyrene	0.90	0.95	0.91	0.91	0.90	0.91	0.91	0.93	0.92	1.8%
Triaccontane	1.01	1.05	1.00	1.01	1.01	1.01	1.02	1.02	1.02	1.5%
Dotriacontane	1.00	1.03	0.99	0.99	1.00	1.00	1.01	1.01	1.00	1.3%
Tetracontane	0.82	0.87	0.84	0.85	0.86	0.86	0.86	0.83	0.85	2.0%

Corresponds with BCMELP Round Robin part 2.1.

EPH in Solids – Method Performance Check Recovery Data										
units = % recovery	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD
Decane	88%	81%	86%	94%	90%	91%	91%	92%	89.1%	4.6%
Naphthalene	90%	86%	90%	94%	92%	94%	94%	96%	92.0%	3.5%
Dodecane	92%	85%	90%	94%	92%	93%	93%	96%	91.9%	3.6%
Hexadecane	93%	90%	96%	97%	97%	95%	97%	98%	95.4%	2.8%
Phenanthrene	93%	88%	95%	94%	97%	96%	98%	98%	94.9%	3.5%
Nonadecane	96%	94%	97%	97%	95%	96%	98%	99%	96.5%	1.7%
Eicosane	96%	96%	97%	97%	96%	96%	98%	100%	97.0%	1.5%
Pyrene	96%	94%	95%	97%	96%	95%	98%	99%	96.3%	1.7%
Benzo(a)pyrene	68%	69%	72%	74%	81%	74%	71%	80%	73.6%	6.5%
Triaccontane	96%	95%	95%	99%	97%	97%	98%	99%	97.0%	1.7%
Dotriacontane	97%	93%	96%	98%	97%	96%	98%	98%	96.6%	1.7%
Tetracontane	99%	86%	99%	99%	97%	96%	99%	101%	97.0%	4.8%

Corresponds with BCMELP Round Robin part 2.2 (spiking procedure was modified; refer to final method document for details).

EPH in Solids – Method Detection Limit Data													
units = mg/kg	#1	#2	#3	#4	#5	#6	#7	#8	Mean	Std-Dev	Target*	Mean Recovery	MDL**
EPHs(10-19)	52.0	51.2	45.7	42.6	42.1	51.4	51.8	37.3	46.8	5.7	44.6	105%	21
EPHs(19-32)	56.1	52.9	54.2	51.3	55.4	55.9	58.4	51.7	54.5	2.4	51.7	105%	9.1

* Target was determined by analysis of spike solution for EPH parameters.

** MDL = Reliable Detection Limit (RDL) at 95% confidence, calculated as (2 x 1.90 x SD) for 7 degrees of freedom.

Note: Data presented demonstrated achievable MDL's. Individual labs must determine their own MDL's. No correspondence with BCMELP Round Robin Study.

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EPH/LEPH/HEPH in Solids - RTC TPH 355-100 Reference Material										
Units = mg/kg	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD
EPHs(10-19)	3273	3333	3508	3444	3393	3526	3471	3481	3429	2.6%
EPHs(19-32)	5382	5186	5356	5105	5217	5380	5327	5317	5284	1.9%
Naphthalene	4.40	4.40	4.87	4.46	4.46	4.69	4.17	4.31	4.47	4.9%
Acenaphthylene*	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	n/a
Acenaphthene*	<3	<3	<3	<3	<3	<3	<3	<3	<3	n/a
Fluorene	2.75	2.77	3.21	2.83	2.87	3.05	3.08	2.82	2.92	5.8%
Phenanthrene	6.81	6.64	7.44	6.67	6.62	7.16	7.03	6.58	6.87	4.5%
Anthracene	0.68	0.61	0.70	0.64	0.63	0.76	0.67	0.63	0.67	7.3%
Fluoranthene	0.21	0.21	0.22	0.20	0.21	0.21	0.21	0.21	0.21	2.5%
Pyrene	0.74	0.75	0.78	0.73	0.75	0.75	0.76	0.75	0.75	1.9%
Benz(a)anthracene	0.087	0.080	0.084	0.083	0.079	0.084	0.084	0.084	0.08	3.0%
Chrysene	0.085	0.088	0.090	0.086	0.087	0.085	0.089	0.089	0.09	2.2%
Benzo(b)fluoranthene	0.047	0.046	0.045	0.043	0.049	0.051	0.042	0.049	0.05	6.7%
Benzo(k)fluoranthene	0.014	0.013	0.011	0.011	0.012	0.012	0.011	0.012	0.01	8.9%
Benzo(a)pyrene	0.051	0.050	0.051	0.054	0.048	0.050	0.049	0.046	0.05	4.7%
Indeno(123cd)pyrene	0.020	0.021	0.021	0.022	0.021	0.022	0.021	0.021	0.02	3.0%
Benzo(ghi)perylene	0.093	0.088	0.095	0.092	0.094	0.095	0.092	0.088	0.09	3.0%
Dibenz(ah)anthracene	0.0067	0.011	0.012	0.013	0.011	0.0062	0.010	0.012	0.01	24.5%
LEPH (calculated)	3262	3322	3496	3433	3382	3514	3460	3470	3417	2.6%
HEPH (calculated)	5381	5185	5355	5104	5216	5379	5326	5316	5283	1.9%

Corresponds with BCMELP Round Robin part 2.3 (EPRM1).

* Values could not be determined due to unresolved interference.

EPH/LEPH/HEPH in Solids - NRC HS3B Reference Material														
Units = mg/kg	#1	#2	#3	#4	#5	#6	#7	#8	#9	#10	#11	Mean	% RSD	Certified*
EPHs(10-19)	473	484	487	483	477	480	402	457	499	442	426	458	7.2%	n/a
EPHs(19-32)	2502	2534	2529	2418	2433	2428	2310	2406	2647	2499	2508	2456	4.0%	n/a
Naphthalene	2.01	1.89	2.00	1.85	1.81	1.77	1.81	1.78	1.76	1.80	1.54	1.82	5.2%	2.14 +/- 0.04
Acenaphthylene	0.60	0.54	0.59	0.59	0.57	0.58	0.98	1.02	0.99	1.04	0.99	0.77	28.5%	0.60 +/- 0.20
Acenaphthene	1.32	1.24	1.31	1.23	1.21	1.25	1.22	1.19	1.15	1.11	0.95	1.20	8.1%	1.25 +/- 0.04
Fluorene	2.09	2.00	2.09	2.00	1.88	1.96	1.87	1.88	1.82	1.88	1.66	1.92	5.3%	2.38 +/- 0.08
Phenanthrene	19.6	18.5	19.6	18.5	17.5	18.2	16.5	16.6	16.0	16.8	15.4	17.6	6.1%	18.8 +/- 0.16
Anthracene	2.69	2.52	2.67	2.58	2.44	2.59	2.51	2.56	2.45	2.59	2.42	2.55	2.8%	2.76 +/- 0.12
Fluoranthene	24.3	22.5	23.6	22.9	22.0	23.0	23.7	24.3	23.9	24.4	23.3	23.4	3.4%	25.33 +/- 0.22
Pyrene	16.7	15.6	16.2	15.8	15.2	15.9	15.4	15.9	15.6	15.9	15.1	15.8	2.1%	18.0 +/- 0.20
Benz(a)anthracene	7.29	6.71	7.03	6.85	6.52	6.86	7.02	7.32	7.31	7.54	7.27	7.07	4.7%	7.91 +/- 0.18
Chrysene	7.86	7.22	7.52	7.35	6.89	7.34	7.72	8.21	8.04	8.22	8.01	7.67	6.3%	8.77 +/- 0.22
Benzo(b)fluoranthene	9.42	8.64	9.18	8.82	8.32	8.98	8.66	8.84	8.91	9.19	8.99	8.90	2.9%	n/a
Benzo(k)fluoranthene	3.72	3.37	3.54	3.42	3.16	3.50	3.24	3.34	3.33	3.45	3.38	3.40	3.3%	n/a
Benzo(b+k)fluoranthene	13.1	12.0	12.7	12.2	11.5	12.5	11.9	12.2	12.2	12.6	12.4	12.3	2.9%	12.8 +/- 0.24
Benzo(a)pyrene	5.52	5.05	5.28	5.15	4.81	5.23	5.20	5.31	5.25	5.46	5.30	5.23	3.6%	5.80 +/- 0.30
Indeno(123cd)pyrene	4.31	3.92	4.23	4.03	3.83	4.07	3.91	3.92	3.80	4.03	3.86	3.99	2.5%	n/a
Benzo(ghi)perylene	3.89	3.54	3.78	3.58	3.42	3.62	3.68	3.65	3.50	3.60	3.48	3.61	2.5%	3.88 +/- 0.30
Dibenz(ah)anthracene	1.09	1.00	1.07	0.99	0.96	1.03	1.12	1.14	1.11	1.18	1.14	1.08	7.4%	0.89 +/- 0.08
LEPH (calculated)	451	464	465	463	458	460	384	439	481	423	409	445	7.3%	n/a
HEPH (calculated)	2451	2487	2480	2370	2388	2380	2263	2358	2599	2450	2461	2426	4.0%	n/a

Corresponds with BCMELP Round Robin part 2.4 (EPRM2).

Data is a composite of 2 batches (batch 1 = samples 1-6, batch 2 = samples 7-11).

* HS3B Certified Values are based on DCM Soxhlet extraction, and are indicated as Mean +/- 2 standard deviations.

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VH in Waters - Instrument Performance Check Relative Response Data									
Units = relative area	#1	#2	#3	#4	#5	#6	#7	Mean	% RSD
Hexane ¹	0.96	1.09	0.96	0.97	0.97	0.96	0.96	0.98	0.3%
Benzene ¹	1.08	1.11	1.08	1.09	1.09	1.08	1.09	1.09	0.1%
Toluene ¹	1.04	1.07	1.05	1.05	1.06	1.05	1.05	1.05	0.1%
Octane ¹	0.98	0.99	0.99	0.98	0.98	0.98	0.98	0.98	0.0%
Ethylbenzene ¹	1.01	1.01	1.01	1.01	1.02	1.01	1.01	1.01	0.0%
m,p-Xylenes ¹	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	n/a
o-Xylene ²	0.95	0.96	0.95	0.96	0.96	0.95	0.95	0.96	0.0%
1,2,4-Trimethylbenzene ²	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	n/a
Decane ²	0.89	0.76	0.88	0.88	0.88	0.86	0.86	0.86	0.3%

Corresponds with BCMELP Round Robin part 3.1.

¹ Relative response calculated against m,p-Xylene.

² Relative response calculated against 1,2,4-Trimethylbenzene.

VH in Waters - Method Detection Limit Data													
Units = ug/L	#1	#2	#3	#4	#5	#6	#7	#8	Mean	Std-Dev	Target*	Mean Recovery	MDL**
VHw(6-10)	24	36	29	31	31	32	29	30	30.3	3.4	25.0	121 %	13

* Target was determined by analysis of spike solution for VH parameters.

** MDL = Reliable Detection Limit (RDL) at 95% confidence, calculated as (2 x 1.90 x SD) for 7 degrees of freedom.

Note: Data presented demonstrated achievable MDL's. Individual labs must determine their own MDL's. No correspondence with BCMELP Round Robin Study.

VH/VPH in Waters - Low Level Spike (2,000 ug/L gasoline)											
Units = ug/L	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD	
VHw(6-oXylene)	851	790	868	947	846	874	869	864	864	5.0%	
VHw(oXylene-10)	171	150	171	171	168	176	174	172	169	4.7%	
VHw(6-10)	1022	941	1039	1117	1014	1050	1043	1037	1033	4.7%	
Benzene	15.3	15.3	15.5	15.3	15.3	15.5	15.5	15.2	15.4	0.8%	
Toluene	101	108	111	107	107	109	109	106	107	3.0%	
Ethylbenzene	20.9	20.3	20.9	20.5	20.1	20.5	20.7	20.1	20.5	1.6%	
m,p-Xylenes	62.8	60.0	61.1	60.0	59.0	60.4	60.1	58.3	60.2	2.3%	
o-Xylene	24.4	23.7	24.1	23.6	23.3	23.7	23.8	23.2	23.7	1.7%	
VPH (calculated)	798	713	806	891	790	820	813	814	806	6.0%	

Corresponds with BCMELP Round Robin part 3.2.

VH/VPH in Waters - High Level Spike (10,000 ug/L gasoline)											
Units = ug/L	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD	
VHw(6-oXylene)	4241	4276	4350	4376	4139	4340	4208	4138	4259	2.2%	
VHw(oXylene-10)	1066	1077	1064	1091	1071	1070	1079	1056	1072	1.0%	
VHw(6-10)	5307	5353	5414	5467	5210	5410	5287	5194	5330	1.8%	
Benzene	76.5	76.5	77.5	76.7	76.3	77.4	77.6	76.0	76.8	0.8%	
Toluene	503	540	556	535	534	546	547	528	536	3.0%	
Ethylbenzene	104	102	105	102	100	102	103	100	102	1.6%	
m,p-Xylenes	314	300	306	300	295	302	300	292	301	2.3%	
o-Xylene	122	119	121	118	117	119	119	116	119	1.7%	
VPH (calculated)	4187	4217	4250	4335	4088	4264	4140	4082	4195	2.1%	

Corresponds with BCMELP Round Robin part 3.3.

Note: BTEX results were obtained from Low Level Spike values x 5, since the High Level Spike was off-scale when un-diluted.

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VH in Solids – Instrument Performance Check Relative Response Data											
Units = relative area	#1	#2	#3	#4	#5	#6	#7	#8	#9	Mean	% RSD
Hexane ¹	0.809	0.809	0.805	0.806	0.805	0.802	0.814	0.817	0.800	0.81	0.7%
Benzene ¹	1.007	1.008	1.008	1.007	1.007	1.005	1.012	1.019	1.003	1.01	0.5%
Toluene ¹	0.995	0.999	0.998	0.998	0.996	0.997	1.000	1.002	0.995	1.00	0.2%
Octane ¹	0.904	0.906	0.904	0.904	0.905	0.903	0.909	0.912	0.901	0.91	0.4%
Ethylbenzene ¹	0.992	0.992	0.992	0.992	0.992	0.992	0.992	0.992	0.992	0.99	0.0%
m,p-Xylenes ¹	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.00	n/a
o-Xylene ²	1.022	1.022	1.022	1.024	1.023	1.021	1.026	1.029	1.022	1.02	0.2%
1,2,4-Trimethylbenzene ²	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.00	n/a
Decane ²	0.957	0.954	0.954	0.955	0.954	0.954	0.960	0.962	0.953	0.96	0.3%

Corresponds with BCMELP Round Robin part 4.1.

¹ Relative response calculated against m,p-Xylene.

² Relative response calculated against 1,2,4-Trimethylbenzene.

VH in Solids – Method Performance Check Sample Recovery Data										
Units = % recovery	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD
Hexane	94%	94%	88%	93%	77%	77%	80%	102%	88.0%	10.4%
Benzene	96%	97%	96%	96%	100%	100%	103%	117%	101%	7.0%
Toluene	95%	95%	95%	95%	101%	101%	104%	111%	99.7%	5.8%
Octane	94%	94%	85%	95%	98%	98%	104%	112%	97.5%	8.1%
Ethylbenzene	95%	95%	95%	95%	102%	102%	106%	108%	99.9%	5.3%
m,p-Xylenes	95%	95%	95%	95%	99%	103%	106%	108%	99.6%	5.4%
o-Xylene	96%	95%	95%	96%	105%	105%	108%	108%	101%	5.9%
1,2,4-Trimethylbenzene	97%	95%	96%	98%	108%	108%	111%	109%	103%	6.7%
Decane	95%	94%	85%	96%	102%	102%	106%	106%	98.3%	7.2%

Corresponds with BCMELP Round Robin part 4.2.

Note: Data is a composite of 2 batches (batch 1 = samples 1-4, batch 2 = samples 5-8).

VH in Solids – Method Detection Limit Data														
units = mg/kg	#1	#2	#3	#4	#5	#6	#7	#8	#9	Mean	Std-Dev	Target*	Mean Recovery	MDL**
VHs(6-10)	37.7	31.3	31.3	31.1	28.6	30.6	32.6	31.1	30.7	31.7	2.5	30.0	106 %	9.3

* Target was determined by analysis of spike solution for VH parameters.

** MDL = Reliable Detection Limit (RDL) at 95% confidence, calculated as $(2 \times 1.86 \times SD)$ for 8 degrees of freedom.

Note: Data presented demonstrated achievable MDL's. Individual labs must determine their own MDL's. No correspondence with BCMELP Round Robin Study.

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VH/VPH in Solids – Low Level Spike (250 mg/kg gasoline)										
units = mg/kg	#1	#2	#3	#4	#5	#6	#7	#8	Mean	%RSD
VHs(6-oXylene)	106.1	111.5	102.7	105.5	108.5	119.7	115.3	117.0	111	5.5%
VHs(oXylene-10)	16.7	19.6	18.9	16.4	17.5	20.2	20.5	18.6	18.6	8.4%
VHs(6-10)	122.8	131.1	121.6	121.9	126.0	139.9	135.8	135.6	129	5.6%
Benzene	2.44	2.55	2.54	2.58	2.50	2.51	2.66	2.68	2.6	3.2%
Toluene	15.4	15.7	15.6	16.0	15.2	15.3	16.5	16.4	15.8	3.1%
Ethylbenzene	3.40	3.48	3.41	3.49	3.37	3.36	3.51	3.56	3.4	2.1%
m,p-Xylenes	10.1	10.5	10.2	10.5	9.84	9.87	10.4	10.7	10.3	3.1%
o-Xylene	3.93	4.13	3.94	4.06	3.86	3.92	4.09	4.15	4.0	2.7%
Styrene	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.01	n/a
VPH (calculated)	87.5	94.7	85.9	85.3	91.2	104.9	98.6	98.1	93.3	7.5%

Corresponds with BCMELP Round Robin part 4.3.

VH/VPH in Solids – Medium Level Spike (1,250 mg/kg gasoline)									
units = mg/kg	#1	#2	#3	#4	#5	#6	Mean	% RSD	
VHs(6-oXylene)	514	566	585	571	544	544	554	4.5%	
VHs(oXylene-10)	97.9	103	99.2	106	92.5	101	100	4.6%	
VHs(6-10)	612	670	684	677	636	645	654	4.2%	
Benzene	12.5	12.4	12.9	12.7	12.5	12.4	12.6	1.6%	
Toluene	80.1	81.3	82.8	81.2	80.8	80.0	81.0	1.3%	
Ethylbenzene	17.5	17.6	18.5	17.8	17.9	17.8	17.9	2.0%	
m,p-Xylenes	52.7	52.6	55.6	53.4	53.8	52.4	53.4	2.2%	
o-Xylene	20.8	20.9	21.3	20.6	21.3	20.7	20.9	1.4%	
Styrene	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	n/a	
VPH (calculated)	429	485	493	491	450	462	468	5.5%	

Corresponds with BCMELP Round Robin part 4.4.

VH/VPH in Solids – High Level Spike (7,180 mg/kg gasoline)										
units = mg/kg	#1	#2	#3	#4	#5	#6	#7	#8	Mean	% RSD
VHs(6-oXylene)	3095	3084	3113	3076	3116	3074	3111	3137	3101	0.7%
VHs(oXylene-10)	533	533	511	505	515	507	517	519	517	2.1%
VHs(6-10)	3628	3617	3624	3581	3630	3581	3628	3655	3618	0.7%
Benzene	79.5	81.5	79.5	81.2	82.2	81.4	81.7	80.8	81.0	1.2%
Toluene	482	494	488	492	496	504	495	504	494	1.5%
Ethylbenzene	102	104	104	105	109	109	106	106	106	2.3%
m,p-Xylenes	307	317	315	320	328	329	320	318	319	2.2%
o-Xylene	119	124	122	124	128	129	126	125	125	2.6%
Styrene	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	n/a
VPH (calculated)	2539	2497	2515	2458	2487	2428	2499	2522	2493	1.4%

No correspondence with BCMELP Round Robin Study.